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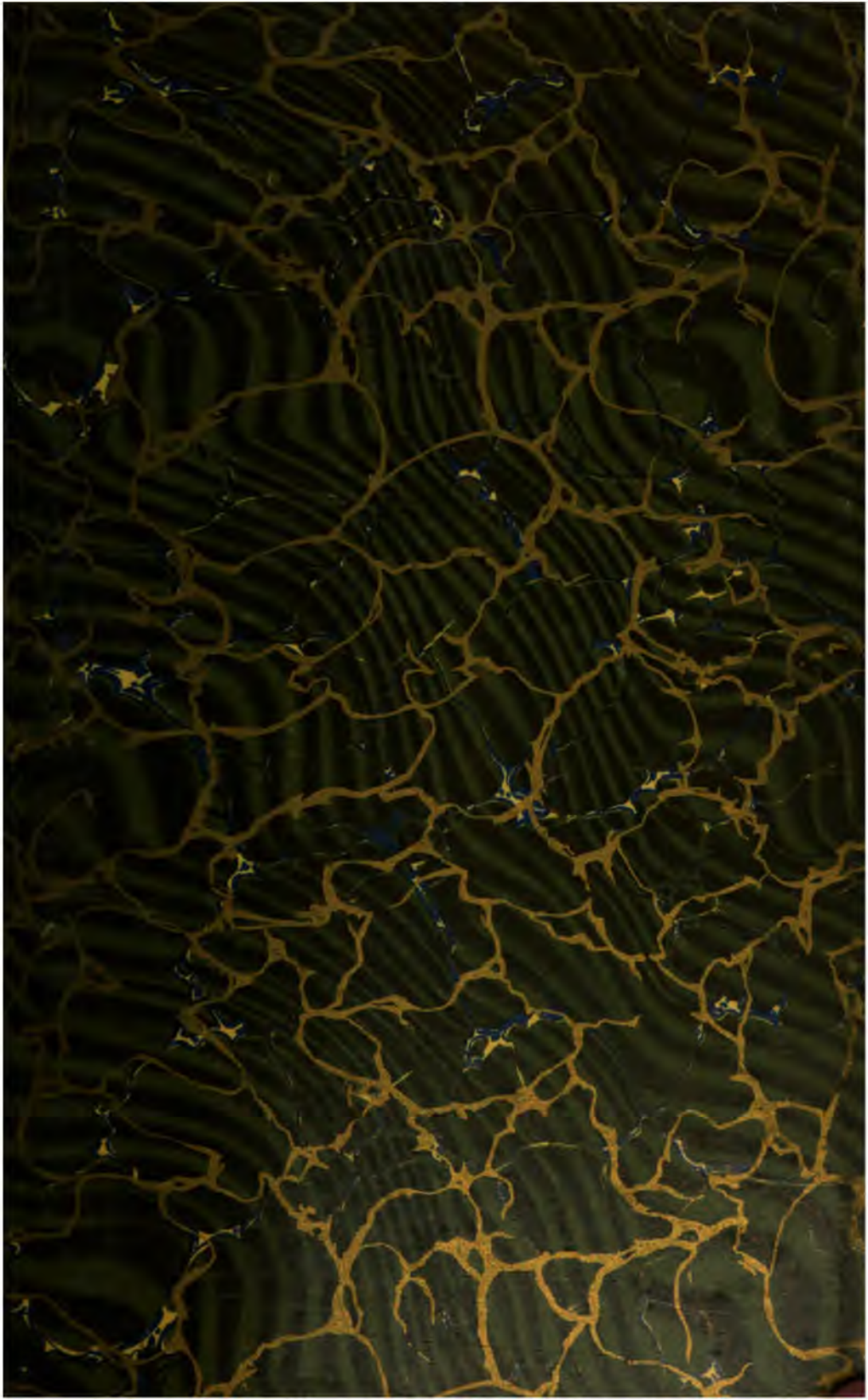
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MANUFACTURE OF SUGAR
COTTONSEED OIL AND PRODUCTS
PETROLEUM AND PRODUCTS
WATER SOFTENING

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PREFACE

The International Library of Technology is the outgrowth of a large and increasing demand that has arisen for the Reference Libraries of the International Correspondence Schools on the part of those who are not students of the Schools. As the volumes composing this Library are all printed from the same plates used in printing the Reference Libraries above mentioned, a few words are necessary regarding the scope and purpose of the instruction imparted to the students of—and the class of students taught by—these Schools, in order to afford a clear understanding of their salient and unique features.

The only requirement for admission to any of the courses offered by the International Correspondence Schools, is that the applicant shall be able to read the English language and to write it sufficiently well to make his written answers to the questions asked him intelligible. Each course is complete in itself, and no textbooks are required other than those prepared by the Schools for the particular course selected. The students themselves are from every class, trade, and profession and from every country; they are, almost without exception, busily engaged in some vocation, and can spare but little time for study, and that usually outside of their regular working hours. The information desired is such as can be immediately applied in practice, so that the student may be enabled to exchange his present vocation for a more congenial one, or to rise to a higher level in the one he now pursues. Furthermore, he wishes to obtain a good working knowledge of the subjects treated in the shortest time and in the most direct manner possible.

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In meeting these requirements, we have produced a set of books that in many respects, and particularly in the general plan followed, are absolutely unique. In the majority of subjects treated the knowledge of mathematics required is limited to the simplest principles of arithmetic and mensuration, and in no case is any greater knowledge of mathematics needed than the simplest elementary principles of algebra, geometry, and trigonometry, with a thorough, practical acquaintance with the use of the logarithmic table. To effect this result, derivations of rules and formulas are omitted, but thorough and complete instructions are given regarding how, when, and under what circumstances any particular rule, formula, or process should be applied; and whenever possible one or more examples, such as would be likely to arise in actual practice—together with their solutions—are given to illustrate and explain its application.

In preparing these textbooks, it has been our constant endeavor to view the matter from the student's standpoint, and to try and anticipate everything that would cause him trouble. The utmost pains have been taken to avoid and correct any and all ambiguous expressions—both those due to faulty rhetoric and those due to insufficiency of statement or explanation. As the best way to make a statement, explanation, or description clear is to give a picture or a diagram in connection with it, illustrations have been used almost without limit. The illustrations have in all cases been adapted to the requirements of the text, and projections and sections or outline, partially shaded, or full-shaded perspectives have been used, according to which will best produce the desired results. Half-tones have been used rather sparingly, except in those cases where the general effect is desired rather than the actual details.

It is obvious that books prepared along the lines mentioned must not only be clear and concise beyond anything heretofore attempted, but they must also possess unequaled value for reference purposes. They not only give the maximum of information in a minimum space, but this information is so ingeniously arranged and correlated, and the

indexes are so full and complete, that it can at once be made available to the reader. The numerous examples and explanatory remarks, together with the absence of long demonstrations and abstruse mathematical calculations, are of great assistance in helping one select the proper formula, method, or process and in teaching him how and when it should be used.

Four of the volumes of this library are devoted to Applied Chemistry. The present volume contains descriptions of the manufacture of sugar, the manufacture of cottonseed oil and products, petroleum and products, and water softening. The manufacture of sugar, both from sugar cane and sugar beets, is rapidly developing and great care has been taken to represent the most modern practice. The cottonseed-oil industry is growing, and there has been a demand for a treatise that considers the subject thoroughly. The addition of rules and regulations of the different exchanges in regard to cottonseed oil and the official analytical methods enhance the value of this treatise. The section on petroleum and products is perhaps the only attempt to treat fully on the fractional distillation, the refining of petroleum, and the manufacture of lubricants and other by-products. Water softening for industrial purposes is one of the economical features of the modern steam plant, and the principles of softening processes, as well as the different types of modern water-softening apparatus, have been fully described and illustrated.

The method of numbering the pages, cuts, articles, etc. is such that each subject or part, when the subject is divided into two or more parts, is complete in itself; hence, in order to make the index intelligible, it was necessary to give each subject or part a number. This number is placed at the top of each page, on the headline, opposite the page number; and to distinguish it from the page number it is preceded by the printer's section mark (§). Consequently, a reference such as § 16, page 26, will be readily found by looking along the inside edges of the headlines until § 16 is found, and then through § 16 until page 26 is found.

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MANUFACTURE OF SUGAR
(PART 1)

INTRODUCTION

1. Definitions.—The term **sugar** was originally employed and intended to classify all substances having a sweet flavor, and thus came to be used almost indiscriminately for cane sugar, fruit sugar, lead acetate (so-called sugar of lead), and other compounds possessing this property. At present, in a general sense, it is reserved almost exclusively to denote crystallized sugar from the tropical cane and the sugar beet, which is *sucrose*, or *cane sugar*. The commercial sugar, whether derived from the tropical cane, the sugar beet, the maple tree, the palm, or some other source, is almost pure sucrose.

2. In chemistry, the word sugar is applied to a large group of compounds of carbon termed *carbohydrates*. These bodies are subdivided into four groups, as follows: (1) **Monosaccharides,** which among other sugars include the glucoses, $C_6H_{12}O_6$, dextrose (also called grape sugar and starch sugar), and levulose (fructose, fruit sugar); (2) **disaccharides,** or sugars, the most important of which is sucrose, or cane sugar, $C_{12}H_{22}O_{11}$; (3) **trisaccharides,** of which raffinose, $C_{18}H_{32}O_{16}$, is of analytical importance, as it occurs in small quantities in the sugar beet; and (4) **polysaccharides,** or starches, $(C_6H_{10}O_5)_x$.

3. The glucoses, dextrose and levulose, are monosaccharides of the hexoses. They occur naturally in plants and are also formed by the hydrolysis of cane sugar. The hydrol-

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ysis is usually accomplished by the action of dilute acids or by ferments on the sugar in water solution. The process is often termed *inversion*. The products of the inversion are equal quantities of dextrose and levulose, and the mixture is called *invert sugar*.

As the word indicates, *hydrolysis* means an absorption of water. Thus, when sucrose is subjected to hydrolysis, the reaction indicated by the following formula occurs:

$$C_{12}H_{22}O_{11} + H_2O = 2C_6H_{12}O_6$$

Chemists have not succeeded in accomplishing the reverse of this reaction; that is, the production of cane sugar from the glucoses.

It will be noted in the study of the processes of manufacture that precautions are always observed to avoid, so far as possible, the hydrolysis, or inversion, of the sucrose by the action of acids or otherwise.

4. History of Sugar.—Etymologically, sugar would seem to be of Indian origin, the earliest forms of the word being *çarkarā* in Sanskrit and *sakkara* in Prakrit. Thence it may be traced through all the Aryan languages, as *shakar* in Persian, *sakkar* in Arabic, *suicar* in Assyrian and Phenician, *saccharum* in Latin, *azúcar* in Spanish and Portuguese, *zuchero* in Italian, *sucre* in French, *zucker* in German, etc.

The precise product indicated by these various names is not always clear, and probably is not identical in all cases. The cultivation of the genuine sugar cane (*Saccharum spp.*) appears to have been common in India and China in very remote times, but there is no documentary evidence on this point earlier than Herodotus. Frequent mention of the "sweet cane" occurs in the Scriptures, but the plant referred to is doubtful. An Indian reed yielding honey is alluded to by Strabo, and a similar statement concerning an Egyptian reed is made by Theophrastus; while Dioscorides actually gives the name *saccharum* to a kind of honey obtained from reeds in Arabia Felix and India; both he and Pliny accurately describe the product as being white and brittle and of a salt-like consistency. Later it seems to have been generally

termed *Indian salt* among the Greeks and Romans, by whom it was obtained in small quantities at great expense from India, and used medicinally.

5. The introduction of cane sugar in the Mediterranean basin must have occurred at an early date; for it was found growing at Assouan, on the Nile, in 714, and was carried into Spain by the Moors in 766, while Sicily engaged in the culture about 1060 to 1090. During the religious wars of the middle ages, the "sweet honeyed reeds," called *zucra*, which abounded in the meadows about Tripoli, were consumed by the Crusaders; and it is evident that sugar making in that neighborhood was conducted in a wholesale and systematic manner. From Cyprus and Madeira, the industry extended, in 1500 to 1600, to most of the West Indies, where it was carried on by Spanish and British colonists; but there is strong evidence in favor of the supposition that several kinds of sugar cane are indigenous both to the West Indies and to almost the whole continent of South America.

6. Establishment of Refineries.—From the extensive growth of sugar in the western tropics there ensued large importations of the raw article into Europe. The introduction of tea and coffee about the same time created a general and wide demand for what had hitherto been regarded as a medicine rather than a nutritive article of diet. Sugar refining appears to have been copied from the Arabs by the Venetians. Refineries were established in England and Germany in the 16th century, and in Holland soon after, and utilized the imported raw sugar.

7. Sugar From Beets.—Up to this time sugar derived from cane was the only kind known in commerce. But, in 1747, Margraf, a German chemist, demonstrated the existence of about 6 per cent. of sugar in beet root; and in 1795, Achard manufactured beet sugar on his farm in Silesia, Germany, and presented loaves of refined sugar to Frederick William III, of Prussia, in 1799. About ten years later, Napoleon used extraordinary efforts to foster the production of native-grown sugar, and grapes, plums, maize, sorghum,

carrots, etc. were also experimented on. The results obtained did not excel those from beets, and the first French factory for making beet sugar was established at Lille, in 1810, by Crespel-Delisse. The sudden and great fall in the price of sugar, caused by the declaration of peace between France and Germany, in 1815, crippled the native industry in Germany as well as in France, but a few of the manufacturers held on tenaciously, and the production of beet sugar, in France alone, rose through many vicissitudes from 1,000 tons in 1827 to 1,075,000 tons in 1906. In Germany, the production in 1906 amounted to 2,400,000 tons.

8. Sugar Production in the United States.—Prior to 1889, the sugar produced in the United States was almost exclusively derived from the tropical cane that grew in Louisiana. The refining branch of the industry depended on foreign sources for nearly its entire supply of raw sugars, which were derived from both beet- and cane-sugar producing countries. Since 1889, and especially since 1899, there has been a large production of beet sugar in America. This sugar is usually ready for consumption when it leaves the factory. The Louisiana factories formerly produced sugars ready for immediate consumption, but at present raw sugar for refining is manufactured almost exclusively. Other American sources of sugar are the maple tree and sorghum. The production from these two sources, however, especially the latter, is of little commercial importance.

9. Chief Sources of Sugar.—From the preceding statements, it may be seen that the world's sugar supply is obtained almost exclusively from two plants—the sugar cane and the sugar beet. Only a comparatively small quantity of palm sugar is produced, and the small quantity of maple sugar made in America is consumed in the raw state.

CANE SUGAR

SUGAR CANE AND ITS COMPOSITION

10. The Plant.—Sugar cane, *Saccharum officinarum*, is a very large grass; its stalk is round and jointed. Normal canes are never hollow. The exterior color varies with the variety. The stalks of the varieties usually grown for sugar production are of a greenish yellow, a purple or purplish-red color, or have stripes of red or purple and green. In certain varieties the green is so light that the canes are often spoken of as being white. The height to which the cane grows varies greatly with the soil and climatic conditions, and ranges from 2 to 6.5 meters (about 7 to 20 feet), seldom attaining the latter height. The cane grown in Louisiana and Texas never reaches full maturity; it attains a height of from 2 to 3 meters (about 7 to 10 feet). The Cuban cane, under favorable conditions reaches a height of from 3.5 to 4 meters (about 11 to 13 feet). The leaves of the sugar cane are ribbed, and, in different parts of the plant, their length ranges from a few centimeters to 2 meters (from about 1 inch to about 7 feet). Sugar cane is a tropical plant, though it is grown successfully in subtropic regions, notably in Louisiana and Texas. Outside of the tropics, it rarely reaches full maturity and produces seed.

11. For commercial purposes, cane is always grown from cuttings, the plantlets sprouting from the eyes at the joints and never from the seed. The stalks reserved for planting are popularly termed "seed." Until within recent years, the true seed of sugar cane was supposed to be infertile. The discovery of canes that were apparently seedlings led to investigations that proved that a part of the seed is fertile. Large experimental plantings of seedlings have been made

with a view to improving the sugar-producing qualities of the cane, and as a result of many comparative trials a few new varieties have been selected and introduced that are large sugar producers. Canes grown from cuttings have all the characteristics of the original plant, while those from the seed usually differ greatly from the parent. So far as is now known, cuttings from all parts of the stalk, when planted, produce canes of equal sugar-making qualities.

12. Planting of Sugar Cane.—The seed bed is usually prepared with great care in Louisiana and in other localities where the conditions are favorable to thorough plowing. This is not the case in many parts of the tropics, and especially in recently cleared lands that cannot be prepared for plowing except at an enormous expense. Where the conditions favor it, the cane is planted in shallow furrows, entire stalks or cuttings being used as seed. In order to secure a good stand of cane, two parallel lines of stalks, laid breaking joints, are often planted. This method of planting in furrows is usually adopted where cane is cut only twice from one planting or where the lands are old. In Spanish-America, especially in recent clearings or in comparatively new lands, the usual method is to plant in small trenches, each about a yard long and a yard apart in the row, a single short cutting of cane being planted in each trench. The cane rows are usually about 6 feet apart.

Cane may be planted at two seasons of the year, namely, spring and fall; or, if in the tropics, at the beginning of the rainy season and near its end. The cane is called *spring* or *fall plant*, according to the time of planting. Whether planted in clumps or in a furrow, the cane soon forms a compact row if the soil is good. A new crop of cane springs up from the stubble left after harvesting, and in the tropics, many crops may be harvested from a single planting. Usually, only two crops are harvested in Louisiana from one planting.

13. Harvesting of Sugar Cane.—Full maturity of the cane is never awaited before beginning the harvesting. It is

essential that the factories have a long working season; hence, just as soon as the stalks contain sufficient sucrose to justify sugar manufacturing, the cutting of the cane begins. The cane reaches sufficient maturity in Louisiana from about October 20 to November 1, and in the tropics from about 6 to 8 weeks after the close of the rainy season. The cane is at its best in Cuba after March 1, and at this time, in favorable localities, the juice contains about 18 per cent. of sucrose and has a coefficient of purity of about 88°, although these figures are often exceeded. The juice from Louisiana cane, even when at its best, does not often contain so much as 15 per cent. of sucrose, and the average analysis for an entire crop seldom shows more than 13.5 per cent. of sucrose.

14. In harvesting the cane, the stalk is first stripped of its leaves by two or three deft strokes with a cane knife having a broad blade; it is then cut down close to the ground, and finally the top is removed at the highest joint that is sufficiently matured. The joint at which to top the stalk is determined by a change to a lighter color. This joint is easily selected in red or stripped canes, but not readily with the extremely light-colored varieties. In Cuban practice, the canes are subdivided into lengths of about 1 yard so as to facilitate loading. After cutting, the canes are thrown into rows or piles, and later they are loaded in carts or cars and hauled to the factory. The loading is usually accomplished by hand, but during recent years mechanical loaders have come into use.

The cane should be ground as soon as possible after it is cut down. This is especially the case in the tropics, where there is always a rapid fall in the coefficient of purity of the juice, with a loss of sucrose, when cut cane is exposed to the sun.

The canes produced from the seed, or cuttings, are termed *plant canes*, and those from the stubbles are called *ratoons*, or *stubble cane*. The ratoons are better sugar producers than plant canes.

The harvesting season lasts from 10 to 12 weeks in Louisiana, and usually about 5 months in the tropics.

15. Composition of Cane and Cane Juice.—The composition of the cane is extremely variable and is influenced largely by the variety, the soil, the climate, and the age of the plant. Stubble canes, or ratoons, usually contain more sugar and woody fiber than plant canes. According to Dr. C. H. Browne, Jr., of the Louisiana Sugar-Experiment Station, the composition of the purple variety of Louisiana cane, as indicated by the results of many analyses at the time of harvesting, in November–December, is as follows:

	Per Cent.
Water	74.5
Ash	.5
Fiber	10.0
Sugars { sucrose, dextrose, levulose }	14.0
Nitrogenous bodies	.4
Fat, wax, pectin (gums), free acids, combined acids	.6
Total	100.0

The reaction of the cane juice with litmus is always strongly acid.

The preceding analysis refers only to that part of the cane harvested for sugar making. The unripe top joints of the stalk are always left in the field.

16. The juice of the cane varies greatly in composition with the part of the stalk from which it is expressed and with the pressure exerted in extracting it. Compared with the upper part, the lower part of the stalk contains a relatively high percentage of sucrose and low percentages of dextrose and levulose. In addition to the sugars, the juice contains mineral (inorganic) salts, nitrogenous bodies (albuminoids, etc.), and non-nitrogenous bodies (wax, fat, etc.).

The sugar content of the juice varies considerably, even in canes grown in the same district. The age of the cane, the

cutting, that is, whether plant cane or ratoons, the soil, the elevation and drainage of the land, and the character of the fertilizers, if used, all affect the sugar content of the juice. Excessive use of a nitrogenous fertilizer produces a rank growth of cane, having a comparatively low sucrose content and an impure juice; whereas, a phosphatic fertilizer is favorable to the production of rich cane with juice of high purity. Cane grown on low land usually contains less sugar and juice of lower purity than that grown on higher lands in the same vicinity.

Mature cane of a good variety contains a high percentage of sucrose and a very low percentage of dextrose and levulose. In such canes, the levulose is often present as a faint trace, or is absent, and the dextrose amounts to only one-tenth or two-tenths of a per cent. In some rare cases, the only sugar present is sucrose.

The relation between the sucrose and the non-sucrose solids of the juice obtained by milling is modified greatly by the amount of pressure applied and the use or non-use of water to saturate the residue of the cane after the first grinding. The juice obtained by light pressure contains less non-sucrose in proportion to the sucrose than that resulting from heavy pressure or from heavy pressure combined with water saturation of the cane residue. Notwithstanding the lower purity of the juice, that is, regarding everything present except sucrose as an impurity, the modern conditions in sugar production demand the highest possible yield of juice in the milling process.

17. Sugars of the Cane.—Except under very favorable conditions of culture, and then only for a short period, the cane contains three sugars, namely, *sucrose*, or *cane sugar*, *dextrose*, and *levulose*. In the cane-sugar industry, the dextrose and levulose are usually grouped together and termed glucose, or invert sugar, the former term being generally employed. The presence of other sugars has been surmised by some investigators. From the manufacturing point of view, however, only the three sugars mentioned are of prime

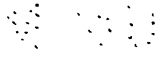
importance, and a knowledge of their properties will therefore be of great practical value to the sugar maker and the chemist.

18. Sucrose, or Cane Sugar.—The sugar known as **sucrose,** or **cane sugar,** forms hemihedral crystals of the monoclinic system. These crystals are transparent and are readily soluble in water and in dilute alcohol. Absolute alcohol, anhydrous glycerine, ether, and chloroform have only a slight, if any, solvent effect on sucrose.

This sugar is rapidly acted on by mineral acids and more slowly by organic acids. This action, as previously mentioned, is termed inversion, or hydrolysis, and the product, consisting of a mixture of equal parts of dextrose and levulose, is called invert sugar. The invert sugar, as well as each of its constituent sugars, is directly fermentable, but sucrose is not. Sucrose must therefore be converted into invert sugar, either by the action of acids or by certain ferments, before it can be fermented.

Sucrose may be inverted by heat alone when in water solution and more rapidly in the presence of air. From this it may be seen that the loss of sugar was great in the old methods of manufacture, in which the juice and concentrated liquors were boiled in the open air. Since the boiling point of the liquid is raised as its concentration increases, the loss of sucrose by inversion is greatest in boiling heavy sirup in an open pan. In modern methods, the liquors are neutralized and concentrated in vacua, thus partly or entirely eliminating losses by inversion from heat and acids. Inversion may occur through the heating of sugar solutions in the presence of inorganic salts and the salts of organic acids, but in the manufacture it is probable that the losses of sucrose from these sources are small. Sucrose, when moist, decomposes at a temperature of 100° C. The heating surfaces of evaporating apparatus should therefore always be kept covered with the sugar solution.

19. Dextrose.—The sugar called **dextrose** forms rhombic crystals when anhydrous, and the hydrated sugar



forms transparent crystals, or crusts. Dextrose is soluble in water and in alcohol, its solubility in the latter depending on the concentration of the solvent.

20. Levulose.—The sugar called **levulose** forms colorless, needle-shaped, rhombic crystals. These crystals are very hygroscopic and are very soluble in water and in alcohol. It is extremely difficult to produce these levulose crystals, and for this reason and owing to the hygroscopic properties of this sugar, invert sugar is frequently termed *incrystallizable sugar.*

21. General Remarks on the Components of Sugar Cane.—A study of the properties of the sugars and other components of the cane is necessary to a clear understanding of reasons for the various steps in the manufacture of commercial sugar. It should be noted that sucrose in water solution is very readily inverted by heat and also by acids; that levulose is easily decomposed by heat and by alkalies; and that dextrose is also decomposed by heat in an alkaline solution, with the formation of highly colored bodies, but resists decomposition more than does levulose.

As will be shown later, both the application of heat and the use of an alkali are necessary in the purification of the juice. Knowing the properties of the sugars, it will be seen that in order to obtain a satisfactory quality and yield of sugar, all highly acid or alkaline solutions must be avoided in the processes of manufacture, as must also excessively high temperatures in treating or concentrating the saccharine solutions in the open air. Unfortunately, to produce the grades of sugar required by the markets, it is not always practicable to work under ideal conditions, with respect to the stability of the sugar. Owing to the tendency of the sugars to decompose and to ferment, cleanliness of tanks and floors is necessary and the work should be conducted with regularity.

The principal component of the juice that must be eliminated in the manufacture is the albuminoid matter. This matter is coagulated at a temperature of about 80° C., but

in the usual defecation process the temperature is raised above 90° C. The coagulated albuminoid matter is somewhat soluble in alkaline juice. As lime is used in the purification of the juice, it is obvious that the reagent must be used with caution.

The ash of the juice contains silica, potash, soda lime, magnesia, iron, phosphoric acid, sulphuric acid, and chlorine. Very little of these substances is removed in the processes of manufacture, and most of them pass into the final residue —the molasses. Part of the acids of the juice is precipitated by the lime in the defecation process, and part remains combined with the reagent in the form of lime salts. Part of the fat, wax, and gums is extracted with the juice in the milling process, and of these substances, the fat, the wax, and to some extent the gums are removed in the defecation process.

COEFFICIENTS USED IN SUGAR MANUFACTURE

22. For convenience and brevity, several expressions, or coefficients, are used by sugar makers and chemists in describing the condition of the juice and products at various stages of the manufacture. These coefficients may be defined as follows:

23. Coefficient, or Quotient, of Purity.—The **coefficient,** or **quotient, of purity** shows the relation between the sucrose and the total solids contained in the material; it is the percentage of sucrose in the total solid matter in the substance. In calculating the *apparent coefficient*, that is, the coefficient ordinarily employed, the solid matter in the juice or in a solution of the material, as indicated by a Brix hydrometer, and the sucrose, as estimated by a direct polarization, are used. The *true coefficient of purity* is the percentage of sucrose in the solid matter, the latter being determined by drying the material.

The true coefficient is used in comparative laboratory tests and in special researches but not usually in conducting the manufacture. The apparent coefficient is meant when the

word "true" is not prefixed, and usually only the word "purity" is used to designate this coefficient. Purities are commonly expressed with the degree mark instead of the percentage sign, or both signs are omitted.

24. The coefficient of purity of cane juice is considered very low when less than 80°, and very high when it reaches 90°. That of the final molasses product is very low in tropical factories at about 30°, and in Louisiana this number often falls below 25°. This difference between the tropical and Louisiana molasses purities is due to differences in the composition of the canes and possibly at times to a decomposition of sucrose.

From these remarks it may be seen that the coefficient of purity is indicative of the sugar-making qualities of the juice, since the higher the purity, the greater the proportion of sugar that must be removed to reduce the molasses to a given purity. In good work, with rich canes, 90 per cent. or more of the sucrose in the juice is recovered in the sugars. The final molasses usually varies but little in purity, whereas the coefficient of the juice often varies greatly. The significance of the coefficient of purity should be thoroughly mastered, since in modern factory methods all the work is conducted with regard to the purity of the materials in process.

25. Glucose Coefficient, or Glucose Ratio.—The **glucose coefficient** is the ratio of the glucose (invert sugar) to the sucrose, multiplied by 100. An increase in this coefficient from one stage of the manufacture to another is usually indicative of inversion of sucrose. This is not necessarily the case, however, since the sugars may not be removed in like proportions in the processes of manufacture, and the mechanical losses may not balance one another.

26. Saline Coefficient.—The **saline coefficient** is the ratio of the percentage of ash to that of the sucrose. It is little used in the cane-sugar industry.

27. Dilution.—By **dilution** is meant the quantity of water added to the normal juice in milling or in diffusion, to increase its weight to that of the diluted juice. The dilution is usually expressed in percentage terms of the weights of the cane and calculated water of dilution.

28. Saturation.—The quantity of water sprayed on the bagasse, in percentage terms of the weights of the cane and water, is the per cent. of **saturation,** or **maceration.**

MANUFACTURE OF CANE SUGAR

29. Synopsis of Manufacture.—The various stages of the manufacture of cane sugar may be briefly outlined as follows: (1) The extraction of the juice—(*a*) milling processes, and (*b*) diffusion processes; (2) the purification of the juice, that is, the defecation and clarification; (3) the concentration of the juice to a sirup; (4) the crystallization of the sugar—(*a*) crystallization in the vacuum pan, (*b*) crystallization at rest, and (*c*) supplemental crystallization with motion; and (5) curing the sugar—(*a*) purging in the centrifugal, (*b*) drying, or granulation, of the sugar, and (*c*) formation of cubes and loaves.

EXTRACTION OF JUICE

30. Processes.—Two processes are used in the extraction of juice from sugar cane, namely, *milling* and *diffusion.* A vast majority of the factories use mills, but very few employ diffusion batteries.

31. Milling Processes.—Mills are usually constructed of three heavy cast-iron rollers that are fitted with strong steel shafts and heavy gearing and housings. The arrangement of a mill with a cane shredder is shown in Fig. 1, and with a Krajewski crusher in Fig. 2. At the left of each of the figures is the mill and near the center and above it, in Fig. 1, is the shredder, and in Fig. 2, the crusher.

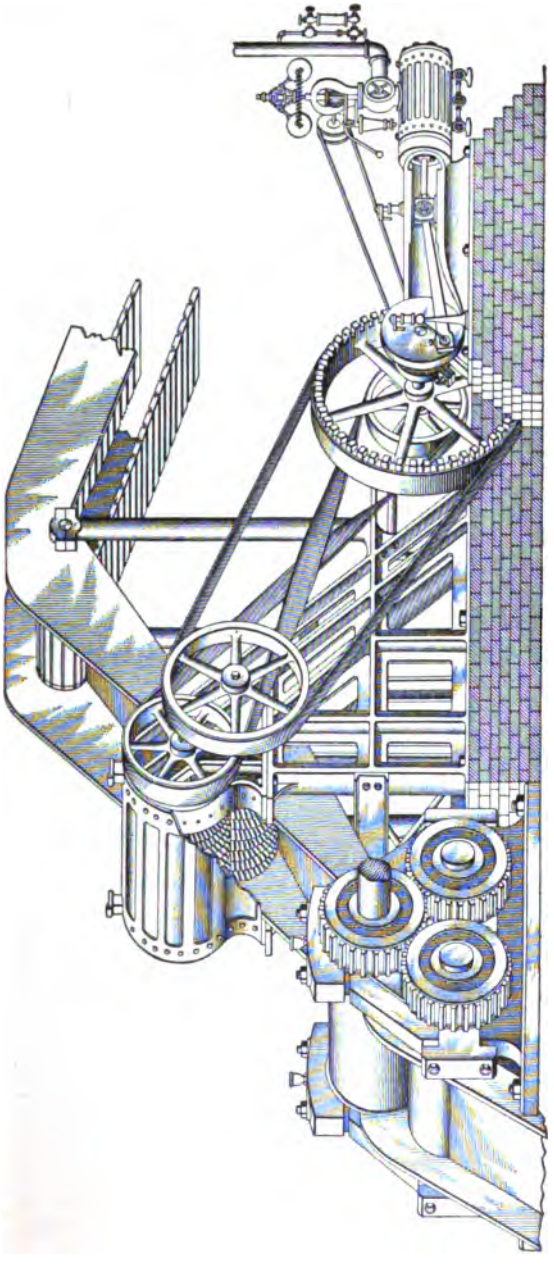


Fig. 1

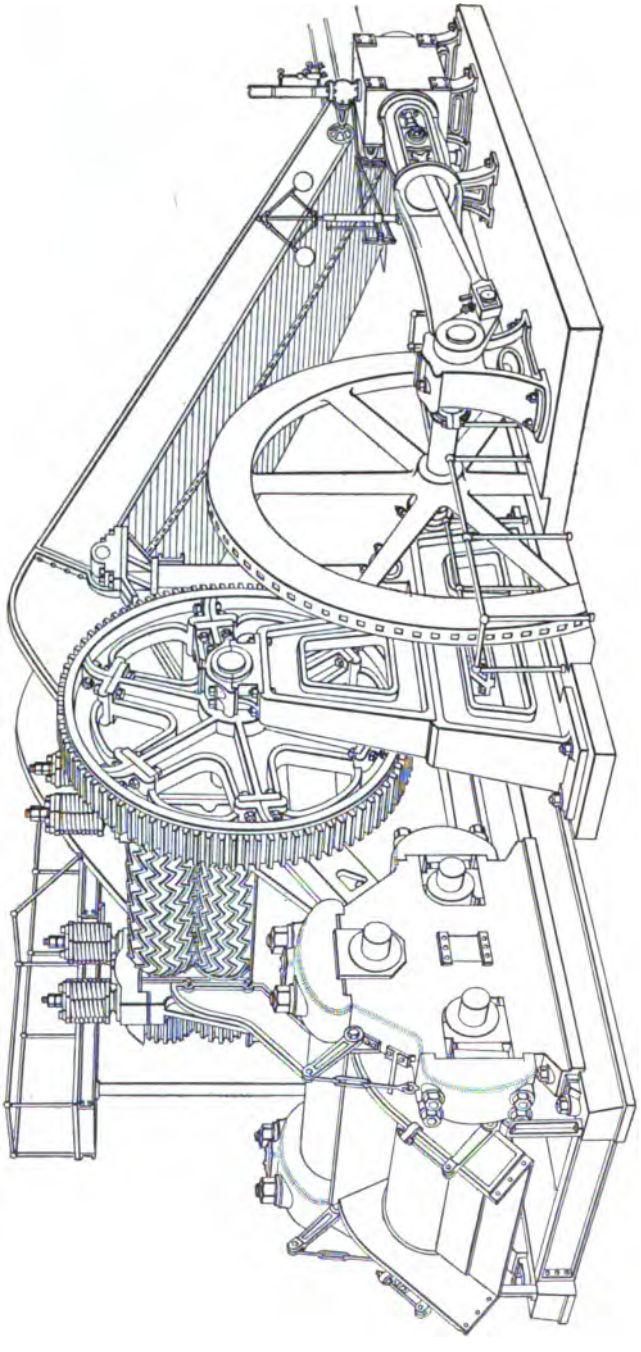


FIG. 2

A chute directs the cane between the top roller and the front, or cane, roller. The partly crushed cane is then forced over a heavy iron plate, called a *turn plate*, or *dumb turner*, and passes out of the mill between the top roll and the back, or bagasse, roll. By means of bolts, which show in the figures at the top and sides of the mill, the rollers are adjusted to give the desired pressure. The space left between the top roller and the cane roller is somewhat greater than that between the top and bagasse rollers. The setting of these rollers is determined by the strength of the mill and the number of mills worked in combination with one another. The pressure on the top roller is often regulated by a hydraulic device, which permits the roller to lift if the feed of cane becomes too thick, or if a piece of iron or other foreign body enters with the cane.

Other combinations of rollers than three, notably two rollers, are occasionally used, but modern plants almost invariably consist of one or more three-roller mills. Instead of using a single three-roller mill, the modern factories usually have two or three mills, and occasionally four mills, driven by one or two engines. The mills are set "tandem," that is, one after another, and the cane passes from mill to mill. The residue, or bagasse, from the last mill of the series is conveyed directly to the fires for the production of steam. This residue is termed *megass* by the English.

32. It is the usual practice to shred, or crush, the cane preliminary to grinding it. Two types of machines are very generally used for this purpose, namely, the *National cane shredder* (Fig. 1), invented by Samuel Fiske, of the Newell Universal Mill Company, the first successful preparatory machine; and the *Krajewski cane crusher* (Fig. 2), of the Krajewski-Pesant Company. The *Marshall crusher*, made by the Whitney Iron Works Company, which is similar to the Krajewski crusher, but differs in the shape of the teeth and certain other details, is also used to some extent.

33. The **National cane shredder**, as shown in Fig. 1, has two cutting, or tearing, rolls made up of toothed steel

disks. The disks of one roll enter the spaces between those of the other, as shown. The disks are driven in opposite directions and at different rates of speed, thus tearing the cane fed to them into shreds, and depositing the shreds in an even layer in front of the mill rolls. The juice that is separated is immediately reabsorbed by the shredded cane. This is not a crushing process.

The **Krajewski crusher**, as shown in Fig. 2, is designed to break the rind and partly extract the juice. The cutting, or crushing, rollers have sharp-edged, wavelike corrugations.

The object of the shredder or the crusher is to relieve the first mill of the strain of crushing the hard rind of the cane. This enables the mills to do more and better work. It also prepares the bagasse to receive water of saturation as will be described later. The crusher also extracts considerable juice.

34. The cane is usually conveyed to the mills on a moving platform called a *carrier*, or *conductor*. It is transferred from the carts or cars by hand or by mechanical devices. In several of the large plants of recent construction, the cane is hoisted from the cars in bundles of 4 or 5 tons and is dropped into a large hopper. Then it is conveyed to the mills by chains, equipped with strong fingers or arms.

The first mill is usually adjusted to extract juice to the extent of about 60 per cent. of the weight of the cane. The second and third mills are set to extract as much juice as their strength will permit. Under these conditions, the bagasse leaves the last mill with a water content of approximately 51 per cent. and containing from 8 to 10 per cent. or more of sucrose, these figures varying with the juiciness and richness of the cane. In order to reduce this great loss of sugar, the bagasse is usually saturated with water after it leaves the first or the second mill, thus diluting the juice it contains before regrinding. This process is termed *saturation*, *maceration*, or *imbibition*.

35. Two methods are commonly used in applying the saturation water, namely, *single* and *double saturation*.

In **single saturation,** the water is sprayed on the bagasse as the latter is expanding on leaving the mill. The water may be applied at the first or the second mill, or at both. The usual method is to apply the water to the bagasse leaving the second mill, as it is then in better condition to receive the water. The juice from all the mills is mixed and is pumped to the defecators for purification.

In **double saturation,** all the water is applied to the bagasse from the second mill. The thin juice from the third mill is strained and is used to saturate the bagasse from the first mill. The mixed juices from the first and second mills are pumped to the defecators.

36. Diffusion Process.—As the **diffusion process** is used exclusively in beet-sugar manufacture, and to only a very limited extent in cane-sugar factories, it will be described fully in the Sections devoted to the beet-sugar industry. A brief outline of the process as applied to cane, however, will be given here.

The process as used in American factories in extracting the sugar from cane is usually maceration rather than diffusion. The cane is reduced to fine shreds, or match-like fragments, and is then introduced into a series of cylindrical iron vessels, where it is systematically soaked in water. The work is so conducted that the first of the series of iron vessels, or diffusers, contains exhausted cane and the last, fresh cane and concentrated juice. The exhausted cane is rejected and juice from the last diffuser is sent to the defecators from time to time, for purification. When a diffuser is empty the succeeding one becomes first in the series, and a freshly filled vessel becomes the last. The liquid near the end of the series of diffusers is heated to a temperature of about 85° C.

Owing to the scarcity of water and the high price of fuel on most plantations and to the difficulty of disposing of the residue, or exhausted chips, this process is little used in cane factories. From the point of view of the extraction of the sugar, however, it is an ideal process.

37. Diffusion of Bagasse.—A diffusion process, or, correctly speaking, a maceration process, for treating bagasse in a diffusion battery has been patented by Naudet. As this process combines with it the purification of the juice, it will be described under that heading in Art. **54.** The maceration of the bagasse in a diffusion battery has been frequently applied.

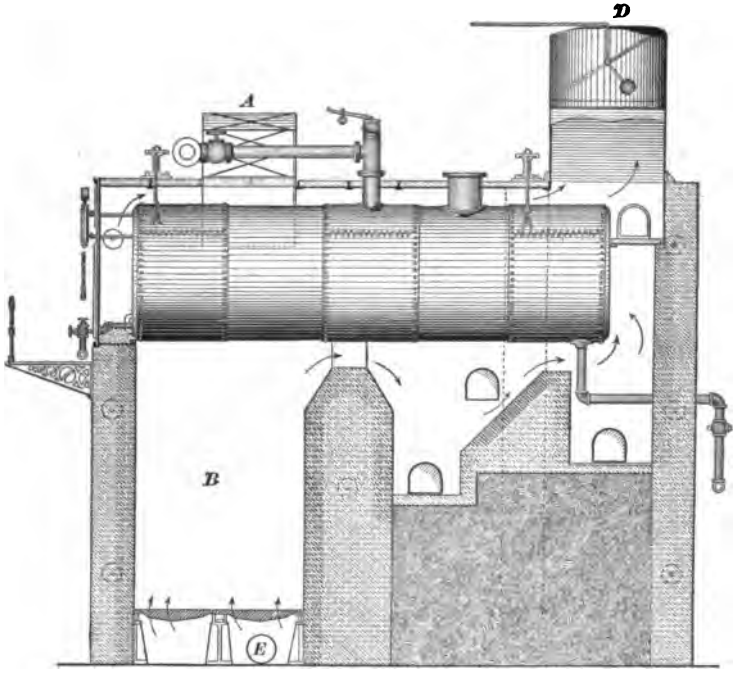


FIG. 3

38. Disposal of Bagasse.—The cane bagasse, under good milling conditions, contains from 50 to 53 per cent. or less of water, and from 6 to 8 per cent. of sucrose. The bagasse makes excellent fuel, but it must be burned in a special type of furnace. Few, if any, factories utilize the bulk of their bagasse for any other purpose.

The furnace used varies somewhat with the type of steam boiler employed. A *Fiske burner*, or *furnace*, Figs. 3 and 4,

with two multitubular boilers, illustrates one of the usual appliances for utilizing the bagasse for producing steam.

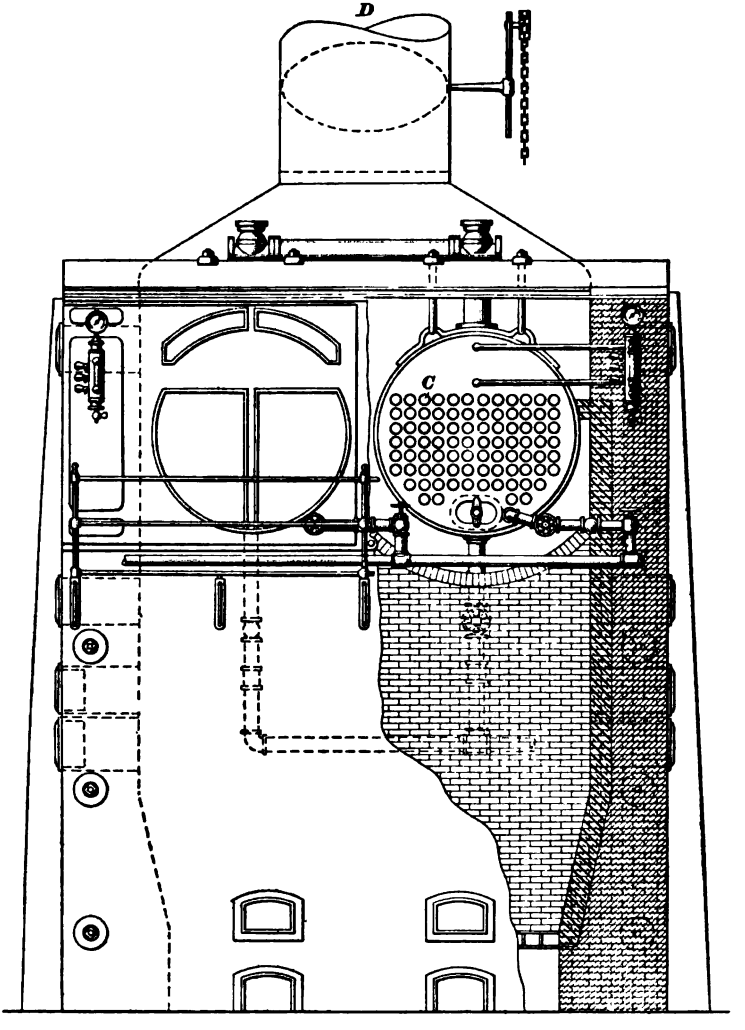


FIG. 4

On leaving the last mill, the bagasse is elevated to the automatic feeder *A*, the balanced door of which opens when

the fuel falls on it and immediately closes after the fuel passes. This automatic feeding of the bagasse is very important, as it is necessary, as far as possible, to avoid the admission of air to the furnace above the grate bars. From the feeder, the bagasse falls between the boiler shells directly on the fire in a large combustion chamber. Air is forced under the grate bars by a pressure blower through the opening *E*. Owing to the nature of the fuel and the large quantity of moisture it contains, the combustion chamber *B* is very large and a very high temperature must be maintained in it. This temperature attained in the combustion chamber is very often 1,300° C. and frequently reaches 1,400° C. On account of this high temperature, it is necessary that the gases have a long distance to travel in contact with the shell of the boiler before entering the flues. With the Fiske type of furnace, the multitubular boilers for a large plant should be about 22 feet long. The heated gases pass under the shell of the boiler, then through the tubes *c*, Fig. 4, to the front end, and then over the boiler to the chimney *d*. Every part of the boiler is exposed to the heat. Owing to the intense heat, a part of the ash fuses to a slag. This slag adheres to the firebrick lining of the furnace and must be removed at intervals.

When water-tube boilers are used, the furnace is usually placed between two boilers.

In the Cook type of furnace, which is also used extensively, the bagasse is burned on a hearth instead of on grate bars, and the air is forced into the burning material through tuyères.

39. Where the installation of machinery in the factory is economical and the cane rich in woody fiber and sucrose, the bagasse furnishes nearly sufficient fuel for the manufacture of the sugar. It is largely supplemented in Louisiana by fuel oil and in the tropics by wood. In modern methods of manufacture, in which only high grades of sugar are produced, more fuel is required than in the production of first sugar and molasses sugars.

The nominal steam-boiler capacity required by a factory varies between very wide limits. It depends on the richness of the cane in sugar, on the grade of sugar made, and on the mechanical equipment and methods of manufacture. A capacity of 700 to 800 nominal horsepower is usually sufficient for a factory that grinds 500 tons of cane in 24 hours.

It has been proposed to use the bagasse for the manufacture of paper stock, and machinery has been installed for that purpose. The results, however, have not been commercially satisfactory.

Small quantities of finely ground bagasse are used in the manufacture of a cattle food called "molascuit," in which the bagasse serves as an absorbent for molasses.

PURIFICATION OF JUICE

40. Juice Strainer.—As it flows from the mill rolls, the juice carries with it a large quantity of fine bagasse, which is termed "trash" or "cushcush." The latter word is used only in the English factories. In well-equipped sugar houses, the juice flows from the pans of the mill on a brass screen that has from 400 to 600 round perforations per square inch. The holes of this screen are prevented from clogging, and the trash is elevated and deposited in front of the second mill by a return-flight, link-belt conveyer, or drag. The flights, or scrapers, are edged with rubber belting.

After being strained, the juice is pumped either to the defecators or to the liming tanks, according to the method of further purification that is used.

41. Defecators and Clarifiers.—The treatment of the juice after being strained depends on whether raw sugar or white sugar is to be made. As comparatively few factories make the latter, the raw-sugar methods will be described first, and later the modifications necessary in making white sugar.

Defecators, as shown in Fig. 5, are usually round iron vessels with double hemispherical bottoms, the inner bottom

being of copper. In heating the juice, steam is admitted to the space between the bottoms. This type of defecator is used almost exclusively in tropical sugar factories.

The American factories use a rectangular iron tank fitted with steam coils for the defecation, and usually term it a **clarifier,** and the process the *clarification.* As will be shown later, the defecation is a distinct process from the clarification. The tropical factories often dispense with the latter process, and those of Louisiana usually accomplish both in the rectangular defecator, or clarifier. In any case, several of these tanks are always used, their number and capacity varying with the size of the factory. As the tanks

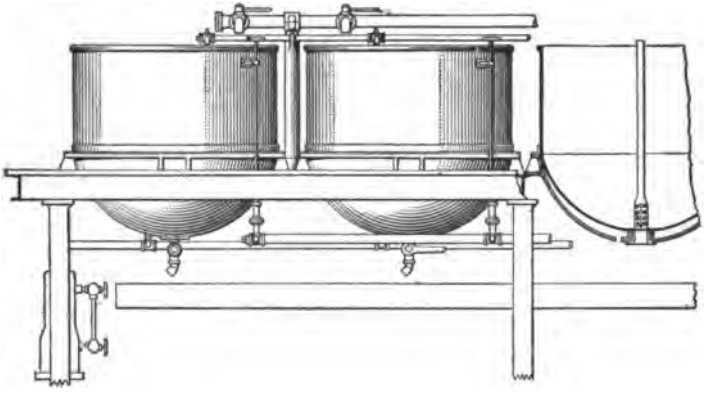


FIG. 5

are used in regular rotation, a sufficient number is installed to allow time for the separation of the impurities and the decantation of the clean juice. Large factories generally employ more than one series of tanks. The rectangular tanks, however, may be readily constructed of sufficient capacity for very large establishments without duplicating the series. Each defecator is provided with an inlet pipe for raw juice; draw-off pipes, cocks, and gutters for clean juice and scums; and a removable perforated plug, to prevent the scum from mixing with the clear juice during the decantation. A steam inlet and an outlet for the water of condensation are also provided. The clarifier differs from the defecator

in having a trough to receive scum that may be brushed from the surface of the defecated juice.

42. A method of defecation and clarification that is extensively used is known as *Deming's system*. This method employs closed steam digesters and closed or open settling tanks of special form. The prominent features of this process are the heating of the juice under pressure to a temperature above its boiling point in the open air, and its constant action, the ordinary process being an intermittent one.

43. A type of clarifier, much used by English factories, and of very efficient construction, is the **eliminator.** This apparatus consists of a rectangular tank in which the juice is heated by a steam manifold having brass or copper tubes. The steam is admitted to the rear end of the manifold, and in boiling the liquor, it throws the scum to the front of the tank. Here, the scum overflows into two scum compartments, which extend parallel with one another across the front of the clarifier. The inner compartment has a row of small holes in the partition wall, through which juice that separates from the scum may flow back into the tank. The heavy particles of scum are either carried by the boiling liquor or lifted by the attendant into the outside compartment, whence they flow through a pipe to receiving tanks, preparatory to filtration.

In the ordinary clarifier largely used in Louisiana, after the heavy scum is removed, the juice is boiled and the scum that rises is brushed into a trough.

44. Ordinary Process of Defecation and Clarification.—The **defecation process,** as the name indicates, consists in separating the impurities of the cane juice that are coagulable by heat and also those precipitable by lime. Of the impurities, the nitrogenous bodies, including the albuminoids, are partly removed by coagulation, and the pectin, fat, and wax, and the mineral and organic acids are precipitated in part by the lime or are removed mechanically by the precipitates.

In using the ordinary defecators, the juice is drawn into the tank until the copper bottom is covered. When the tank is filled, sufficient lime, in the form of a milk, is added to render the entire charge slightly alkaline, and steam is then admitted to the heating chamber between the double bottoms. The admission of steam is so regulated that when the juice reaches a point about 6 inches below the rim of the tank, its temperature is only a few degrees below its boiling point. The stream of juice is now turned into the next defecator of the series. The heating of the juice in the first tank is continued until the gases liberated just break the heavy surface scum, or "blanket." This is technically termed the *cracking point*. At this stage, the steam is shut off from the defecator and the juice is left at rest, so that the heavy precipitates will sink to the bottom of the tank and the flocculent matter will rise to the surface of the liquid.

If the proper proportion of lime has been used, the precipitates will form a compact mud on the copper bottom, the juice will soon separate in part from the blanket, leaving the latter firm and porous, and the juice between the mud and the blanket will be bright and sparkling. The impurities will usually settle well even if a considerable excess of lime has been used; but insufficient lime, on the contrary, produces a turbid, or cloudy, juice. In raw-sugar manufacture, the lime should be added to slight alkalinity; but in making white sugar, the limed juice should be faintly acid to litmus paper. The precipitates and flocculent matter separate well from both alkaline and slightly acid juice. The sugar maker usually judges the quantity of lime to use by the indications already described and by the appearance of the scum and the rate at which the floccules move in a test sample of the defecated juice. By these indications, he is guided in liming the next and subsequent defecators of juice.

45. The quantity of lime to be used in making raw sugar may be estimated by the following simple chemical test: To a small portion of defecated juice that has been filtered through paper, add a solution of saccharate of lime,

a few drops at a time, noting whether or not a precipitate forms. A glass test tube should be used in this experiment. If a precipitate appears, the juice has not received sufficient lime; if no precipitate forms, either too much or just enough lime has been used in the defecation. If a precipitate forms or does not form, the liming of a subsequent defecator of juice should be changed accordingly, and the test should be repeated. The addition of lime should be continued as long as a precipitate appears.

46. After allowing sufficient time for the separation of the impurities, the draw-off cock at the bottom of the defecator is opened and the cloudy juice is run into a suitable gutter leading to the scum tanks. When the clear juice follows, it is run into a trough leading to the clarifiers or storage tanks, and, finally, when the juice becomes turbid, the central plug is removed and the mud is diverted into the scum canal.

Where the combined defecators and clarifiers are used, the procedure is the same as already described, except that the blanket scum is removed with a paddle, called a *brush*, and the juice is boiled and skimmed, that is, clarified. After this treatment, the juice is allowed to remain for some time in the clarifier, which is used in lieu of a settling tank, or subsider, and is then decanted into the storage tanks.

47. In the manufacture of raw sugar, the juice in certain factories is limed in excess and then passed through a sulphur box or other sulphuring device, where sufficient lime is precipitated by the sulphurous acid to reduce the alkalinity of the juice to that in the ordinary process. It is claimed that this process yields very pure, free-boiling juices, and that the molasses has less viscosity than would otherwise be the case.

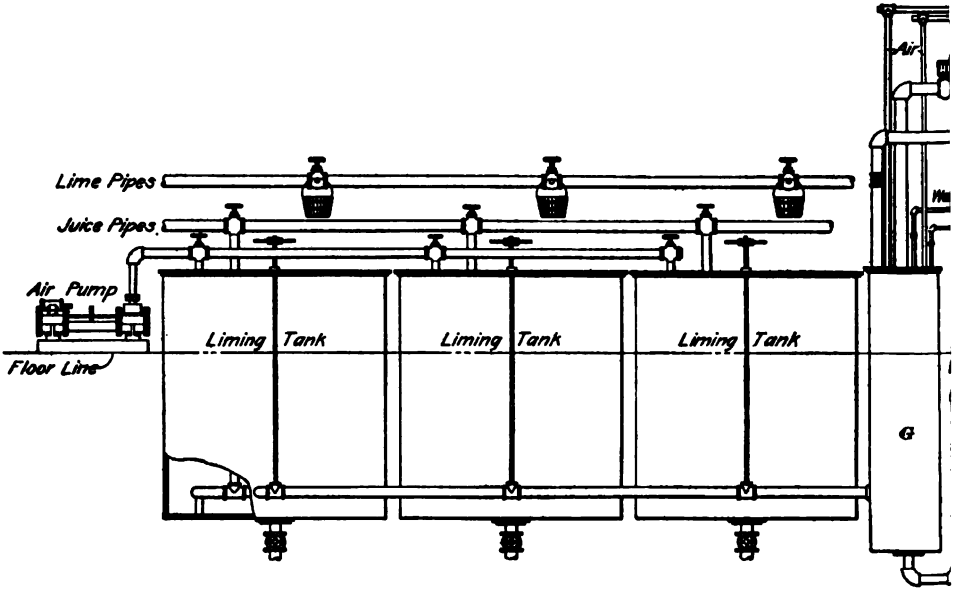
In producing white sugars, the cold juice, just as it flows from the strainer, is saturated with sulphurous-acid gas, is then limed to nearly neutrality to litmus paper, and the defecation is conducted as already described. This process is also used to some extent in making Louisiana raw sugars, but

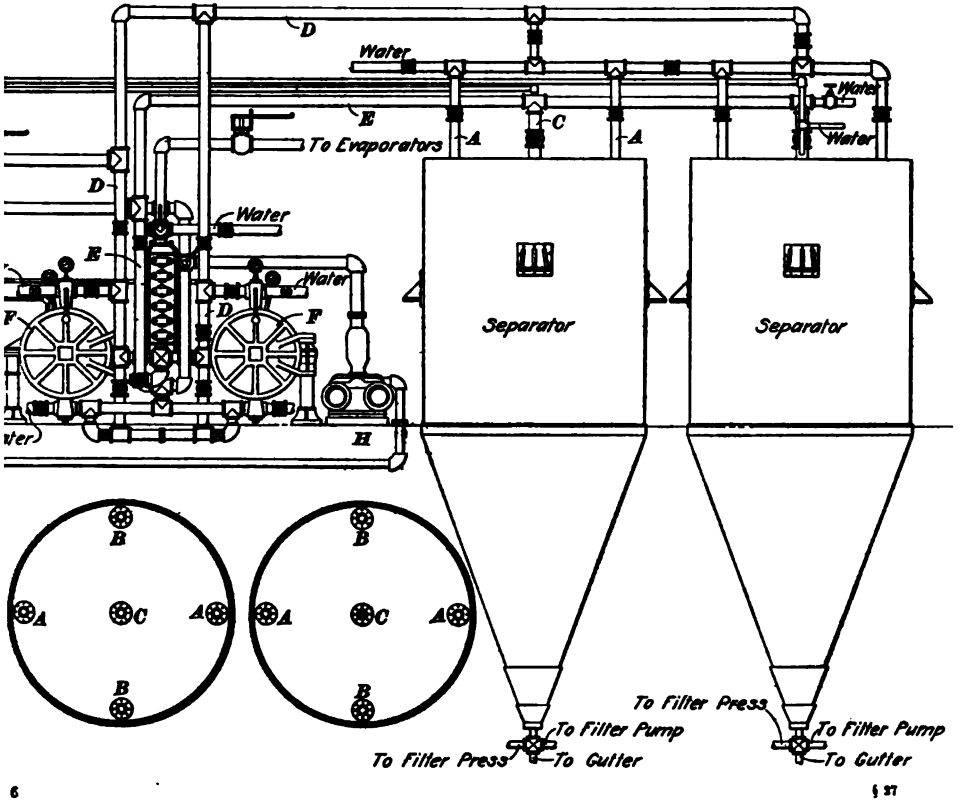
where so employed in the tropical factories, lime is added to the juice to alkalinity. Phosphoric acid is occasionally used in the manufacture of white sugar. The juice is limed in the defecation to slight alkalinity to litmus paper. After the removal of the scum, by decantation or otherwise, sufficient phosphoric acid is added to neutralize the excess of lime, and the juice is then brushed or clarified.

In still another modification of the defecation process, the juice, after liming, is heated to near its boiling point in a tubular heater. It is then run into an ordinary defecator, where the heating is continued until it boils. In this process the separated impurities settle to the bottom of the defecator, instead of remaining partly at the surface. This behavior of the precipitates is due to the expulsion of the gases from the juice during the boiling.

48. Deming's System of Defecation and Clarification.—The system of defecation and clarification known as **Deming's method,** is the only radical change that has been made and extensively applied in the purification of cane juice since the early days of the cane industry. In this method, the juice is limed in the cold in a series of tanks shown at the left in Fig. 6. At the right of the group of liming tanks and connected with each by suitable pipes and valves is the pump tank *G*, which is deeper than the other tanks and into which the latter may be drained. The lime, in the form of a milk, is added to the juice from time to time, as the liming tank is filling. Agitators, not shown, prevent the lime and precipitates from settling to the bottom of the tank. The air pump shown in the figure is used in many installations to force air through perforated pipes into the juice and thus agitate the latter. The quantity of lime to be used is ascertained from an examination of the clarified juice, as in the ordinary process.

In addition to the liming tanks, a digester, an absorber, and a series of settling tanks are required. The digester is often installed in duplicate so as to facilitate the cleaning of its tubes or to increase its heating surface.


Air
Lime Pipes
Juice Pipes
Air Pump
Liming Tank
Liming Tank
Liming Tank
Floor Line
G


D
Water
E
To Evaporators
A
C
A
Water
Water
D
Water
E
Water
F
D
F
Separator
Separator
H
B
A
C
A
B
B
A
C
A
B
To Filter Press
To Filter Pump
To Filter Press
To Gutter
To Filter Pump
To Gutter

The digester is a cylindrical iron vessel fitted with copper heating tubes. In the figure, the digesters in duplicate are shown at *F*. The absorber is located between the digesters and is composed of a system of wrought-iron pipes, in each of which is a copper tube. The iron and copper pipes have suitable head-pieces and return bends for the circulation of the juice. In operating the apparatus, the juice is pumped by means of the juice pump *H* from the liming tanks, around the copper tubes of the absorber, and then through the tubes of the digester, where it is heated by steam to a temperature of 112° C. or even higher. From the digester the juice returns to the absorber, passing through its tubes. The function of the absorber is to reduce the temperature of the juice to below its boiling point in the open air, thus preventing it from flashing into steam when the pressure is reduced. In this way, the absorber tends toward economy in fuel. It is usually sufficient to pass about three-fourths the total volume of the cold juice through the absorber, so as to reduce the temperature of the clarified juice to below its boiling point. The apparatus should be operated at a minimum juice pressure of 25 pounds, as indicated by a gauge. As the heating surfaces foul, the pressure rises, but it should not be allowed to exceed 50 pounds.

49. From the absorber, the juice flows into conical-bottomed settling tanks, or separators, shown at the right in Fig. 6. The settling tanks shown here illustrate a recent improvement in this apparatus. In this case, the tanks are closed, and they are termed "separators" by Deming. Inside of each tank is a chamber shaped like the frustum of a cone, but open at both top and bottom and with the large opening below. The settling tanks are arranged in series. The clarified juice passes into the outer chamber of the first, or primary, tank, flows downwards to the inner chamber, and then upwards and through the overflow pipe to the secondary settling tank. As the surface area of the juice in each chamber is large in proportion to the quantity of juice flowing through it, the current is slow. The descending

juice carries the precipitates with it, and as it rises in the inner chamber to the overflow, it leaves a large part of them in the conical bottom. The flow of juice through all parts of the apparatus is constant while the mills are in operation. From time to time, a cock at the lower part of the settling tank is opened and the mud is drawn off into the scum tanks.

On leaving the digesters, the juice, under pressure, passes through the pipes *D* and *A* into the separators and flows from these to the pipe absorber through *C* and *E*. From the absorber it passes to the charge tanks of the multiple-effect evaporator. The temperature of the juice is reduced to about 82° C. in the absorber.

50. Carbonatation Process.—In treating cane juice, a modification of the **carbonatation process** used in beet-sugar factories is applied extensively in Java, but in other places it is used only to a very limited extent. This process gives excellent results from a sugar-making point of view, and its limited application is due only to the high working expense.

As this process is the only one used in beet-sugar manufacture, it will be described fully later. Only the modifications necessary in its application to cane sugar will now be considered. The carbonatation may be done by either the *single* or the *double method.*

51. Single Carbonatation.—In the **single-carbonatation method,** the juice is rendered strongly alkaline with a quantity of milk of lime equivalent to about 1.25 per cent. of its weight of quicklime. A smaller quantity often suffices. The lime is then saturated with carbolic acid. During the carbonatation, the temperature of the juice is maintained at or below 60° C., but as soon as the lime has been completely precipitated, it is quickly raised to above 90° C. and the juice is then filtered.

52. Double Carbonatation.—In the **double-carbonatation process,** the lime is added in two portions. After the first liming, carbonic acid is forced into the juice until the alkalinity of the latter is approximately equivalent to

1 gram of quicklime per liter, a temperature not exceeding 60° C. being maintained throughout the operation. After freeing the juice from the precipitate by filtration, it is again limed, but only a very small proportion of the reagent is used. After the second liming, the juice is again carbonated and further treated, as in the single-carbonatation process.

The temperature is kept at 60° C. or lower while the juice is strongly alkaline. This is done to prevent the decomposition of the invert sugar and the consequent formation of highly colored products. The presence of an excess of lime at the end of the first carbonatation, in the second process, is to prevent a part of the precipitate from being redissolved. The double carbonatation yields purer juices than the single process.

53. In the manufacture of white sugar by the carbonatation process, after the nearly complete precipitation of the lime, the juice is "sulphured," that is, treated with sulphurous acid, until its reaction is faintly acid to litmus.

Carbonated juices, as well as those sulphured after a single or double carbonatation, may be readily filtered through filter presses. For the filtration of juices clarified by the ordinary processes, mechanical filters that work under low pressures are necessary. Mechanical filters will be described later. The clarified cane juice is not usually filtered.

54. Naudet Process of Extracting and Purifying Juice.—In the **Naudet process,** which is controlled by the Harvey Engineering Company, of Glasgow, the extraction of juice from the bagasse and the purification of juice from both the cane and the bagasse are carried on simultaneously.

The cane is first crushed or shredded and passed through the first mill, as previously described in connection with the milling processes. The bagasse from this mill is extracted in a diffusion battery of eight cells, first with very hot juice and then with water. The diffusion battery is similar to the battery that will be shown later, in *Manufacture of Sugar*, Part 3, though the cells may be arranged in a circle or straight line, as may be convenient, and the circulation of

the juice in the last vessel is accomplished by means of a pump.

55. In conducting the Naudet process, diffuser No. 1 is filled with bagasse; then water is circulated through it and the heater, until the thin juice formed acquires a temperature of from 200° to 220° F., when it is forced into a mixing tank. In the meantime, diffuser No. 2 has been filled with bagasse and the thin juice from the mixing tank is circulated through it, as in the case of the previous vessel. While heating the juice and exhausting the bagasse in No. 2, water is passed through No. 1 and into the mixing tank, so as to supply any deficiency there may be in the quantity of thin juice and to wash the partly exhausted bagasse. These operations are repeated with fresh bagasse in diffuser No. 3, the water passing through Nos. 1 and 2 to the mixing tank, and so on until six diffusers are in operation. The object of these manipulations is to extract the sugar from the bagasse.

Assuming that a measuring tank has been filled with normal juice from the mill and that diffuser No. 7 has been filled with bagasse, the normal juice is run into No. 7 and half fills the diffuser. The juice in the mixing tank is limed and is run into No. 7 until the cell is completely filled; and it is then circulated through this diffuser by the pump until its temperature is raised to about 200° to 220° F. During this operation, the juice is defecated and the precipitates are filtered out of it by the circulation through the bagasse. As soon as the desired temperature has been reached, a measured quantity of juice is drawn from diffuser No. 7 and is sent directly to the evaporators to be concentrated to sirup. The circulation of the juice is forced by compressed air or by water pressure in drawing the juice. When the measured quantity has been drawn, the current is diverted into the mixing tank, to be used in diffuser No. 8. While No. 8 is filling with bagasse, No. 1 is cut out of circulation and opened, and the exhausted bagasse that it contains is discharged on a carrier and conducted to a second mill, where it is pressed preparatory to use as fuel. The water

expressed from the bagasse contains only a trace of sugar and is run to waste. As in the diffusion process, each time that a charge of juice is drawn, the first diffuser of the series is cut out and the exhausted bagasse is removed.

56. Remarks on Defecation and Clarification Processes.—Many processes have been devised for the purification of cane juice, but only those that have been described are in use. In very small factories, an open fire is often used instead of steam in the defecation, clarification, and concentration of the juice, all of which are accomplished in open kettles.

The methods that have been used and discarded include, among others, the following: processes employing clay or alum to increase the rate of the settling of the precipitates; bisulphite of lime, for bleaching and precipitation; albumen, to promote flocculation; also, electrolytic processes and several processes employing bisulphites and hyposulphurous acid.

57. The clarified juice is far from being a solution of sugar and water. Many of the impurities cannot be removed from the juice except by processes that are not commercially profitable. The impurities that remain in the juice impede the crystallization of the sugar and are the cause of the final molasses by-product. The impurities also separate from the liquors to some extent during the concentration and thus foul the heating surfaces of the evaporators. The heating surfaces of the defecators and clarifiers also become foul, the scale formed consisting largely of lime compounds and silica. As the scale is a poor conductor of heat, the surfaces must be cleaned frequently.

The copper surfaces of the double-bottomed defecators may be cleaned by scouring. This and other types of defecators are more readily cleaned, without delaying the manufacture, by covering the copper surfaces with water that is strongly acidulated with muriatic acid. The acidulated water should be boiled for a few minutes, and then run into the sewer. From 4 to 6 pounds of the commercial acid is required to clean a 700-gallon, double-bottomed defecator.

58. Chemical Reagents Used in Purifying the Juice.—*Lime* is used in all the processes now employed in purifying cane juice. The following methods are used in preparing the lime:

1. Quicklime is ground to an impalpable powder, sifted, and stored in covered casks, or barrels. This lime should be slaked in water immediately before using it.

2. Quicklime is heaped on a floor, and then sprinkled with water so as to slake it and produce a dry powder. For use, this powder should be sifted and reduced to a milk, as required, with raw juice.

3. Milk of lime, reduced to 18° Baumé, is circulated through pipes by a special pump and returned to the pump tanks. This circulation of the liquid keeps the lime in suspension. The milk is drawn off where required, from branch pipes, and a measured quantity is used in liming the juice.

4. A quantity of quicklime is slaked and reduced to a milk, and the liquid is strained into a box or a tank. After allowing sufficient time for the lime to settle, the supernatant lime water is drawn off, leaving a thick lime paste. Weighed portions of the paste are diluted with juice, as required, for use in the defecation.

When dry lime is used, whether lime powder or dry slaked lime, the charge for each defecator should be weighed and not measured.

59. *Sulphurous acid* is always used in the gaseous state in cane-sugar factories. This acid is produced by the combustion of sulphur in the air, in a stove designed for that purpose. The stoves employed are of two types, namely, *closed*, into which air is forced by a pump; and *open*, into which air is drawn by natural draft, by the juice pump, or by a steam ejector. The first form is seldom used in the cane-sugar industry, being employed almost exclusively in beet-sugar manufacture.

60. The open type of stove is of cast iron and is usually shaped like an inverted letter **U**, in cross-section. A large cast-iron pipe leads from the dome of the stove to the saturating

device. Both the stove and the pipe should be water-jacketed. The sulphur is burned in a pan at the bottom of the stove, and the quantity of air admitted is regulated by a damper. The saturating apparatus is often a pump, which draws sulphurous-acid gas from the stove and juice from the mill tank and then mixes and delivers the two at the defecators.

A very common saturation device consists of a tall wooden box or column fitted with either slanting or perforated shelves. The mill pump delivers the juice into the top compartment of the box, from which it falls in a shower or series of cascades to the bottom of the column and is carried by a pipe to the defecators. At the same time a current of sulphur fumes is drawn into the lower part of the box and travels upwards, meeting the shower of juice and being largely absorbed by it. A flue at the top of the box carries off the surplus fumes and air. This flue may be made of either wood or lead. If the natural draft is not sufficient, a small steam nozzle directed upwards in the flue will produce the desired result. The nozzle should be of lead hardened with antimony.

61. Sulphurous acid bleaches the coloring matters of the juice and in doing so is partly oxidized to sulphuric acid. In common with other mineral acids, it has the property of coagulating albuminoids. If juice acidulated with this acid is heated or is is not promptly neutralized, a loss of sucrose results through inversion. This reagent has almost entirely superseded animal charcoal in the production of white sugar in cane- and beet-sugar factories.

FILTRATION OF JUICE AND SCUMS

62. Filtration of Juice.—Filter presses and filters will be described later in connection with beet sugar and sugar refining. Clarified juice is difficult to filter, except when obtained in connection with the diffusion process or by carbonatation. Clarified diffusion juice may be strained

through bag filters, such as are used in refineries. Carbonatation juices filter readily through all types of filters. Juice defecated and clarified by the ordinary process filters with great difficulty through cloth in filter presses. The minute particles of almost invisible flocculent matter suspended in the juice soon coat the cloths and prevent filtration. The defecated juice, however, may be filtered with very low pressure through cotton cloth or sand in the so-called mechanical filters. In the cloth filters of this type, the mud collects on the outside of the bag and remains soft and pervious, falling from the cloth from time to time as it accumulates.

Fine bagasse is used to a limited extent as a filtering medium. The bagasse soon clogs with the flocculent matter of the juice and is then returned to the mills to be reground with other bagasse.

63. Filter Pressing of Scums and Precipitates. The scums and precipitates from the defecation and clarification are collected in blow-up tanks preparatory to filtration with presses. These tanks are fitted with perforated pipes, for blowing steam into the scums, and also with cocks at different levels, for decanting the clear juice that separates from the material after heating it.

In the manufacture of either raw or white sugar, it is often advisable to lime the scums to alkalinity, and then heat them with the blow-up pipes to the boiling point. After heating, the contents of the tanks should be left at rest, for the subsidence of the precipitates and the subsequent decantation of the clear juice. This treatment promotes the filtration and reduces the loss of sugar in the press cake. The fine flocculent matter in the thin scums sometimes forms an almost impervious coating on the filter cloths and greatly reduces the capacity of the presses. In the material prepared by liming, heating, and decantation, the porous blanket scums and the heavy precipitates form an excellent filtering medium, and no trouble is experienced in the filtration. Some factories, however, do not prepare the scums as

just described, but in many instances this method has given satisfactory results.

The scums may be forced into the press either by a *monte jus*, that is, by direct steam or air pressure, or by a pump. The latter, however, is preferable. The pressure in the press should not exceed 60 pounds, while a much lower pressure is usually sufficient. In factories having enough tank and evaporator capacity, a saving of sugar is effected by diluting the scums with water after draining off as much juice as possible.

64. Filter presses are usually arranged so that the washing of the residue, or press cake, may be done systematically by passing water through the cake and thus driving out the juice it contains. This method demands very large filter-press capacity, however, and is seldom used except in beet-sugar factories.

The filter-press work is frequently seriously impeded by mixing the precipitate or mud that collects in the sirup tanks with the scums. It is preferable to mix this precipitate with the raw juice before defecation. Where clarification follows the defecation and the process is carried on in separate tanks or in combined defecators and clarifiers, the mud washed from the sirup tanks may be returned to the clarifiers. Care, however, should be taken that this material is largely diluted with juice, otherwise difficulty may be encountered in separating the mud from the clear juice in the subsiders. Should the mud tend to rise to the surface of the juice during settling, the surface of the juice should be liberally sprayed with cold water, which, in falling to the bottom of the tank, usually carries the flocculent matter with it.

With the Deming process of clarification, the sirup-tank washings, or "tank bottoms," as such material is usually termed, should be gradually run into the pump tank to be reclarified. Where it is impracticable to treat the tank bottoms as recommended, they should be run into a separate blow-up tank and then heated to the boiling point. After

settling, the clear liquor should be decanted. Liberal quantities of water should then be used in diluting this material, preparatory to steaming and filtering it. The juice from the filter presses is usually pumped directly to the charging tanks of the evaporators. Where practicable, the juice should first be reclarified, because it is often cloudy, owing to a defective or badly adjusted cloth. Each filter plate of a press is provided with a juice cock for disconnecting it in the event of a cloth breaking. When a cloth is badly adjusted, the press must usually be opened and dressed with clean cloths. This, however, involves loss of time and additional labor for washing the cloths; therefore, when possible, it is preferable to let the press fill slowly under very low pressure. While this may result for a time in a cloudy filtrate, the opening at the defective cloth often fills with the precipitate, permitting the filtration to proceed as usual.

The filter cloths may be used several times without being washed, provided the press is completely filled with well-pressed cake. The latter should leave the cloth freely, soiling it but little. The workman can tell when to open a press by noting whether the juice dripping from the cocks has almost completely ceased, or by closing the inlet valve and drawing the mud from the channel and then noting its volume. If the volume of this mud is greater than that of the channel, it is evident that the press is not full of hard cake. The filter cloths are washed and dried by means of the appliances used in large laundries.

MANUFACTURE OF SUGAR

(PART 2)

MANUFACTURE OF CANE SUGAR
(Continued)

CONCENTRATION OF JUICE TO A SIRUP

1. The juice having been freed from impurities, so far as the usual methods of manufacture permit, it is concentrated to a sirup, preparatory to a final concentration with crystallization of the sugar, in a vacuum pan.

The evaporation of the juice was formerly conducted in open vessels that were worked in batteries, or trains. Apparatus of this class is now used only in very small factories and especially in those located in remote tropical regions, where local market and transportation conditions render work on a small scale profitable.

2. Evaporation in Open Kettles and Steam Trains. A battery of open kettles is illustrated in plan and in section in Fig. 1. The kettles c, d, e, f are arranged over a furnace and flue, each at a level somewhat lower than the one preceding it. The kettles a, b near the chimney are used as clarifiers, from which the juice is drawn into kettle c. The entire series of vessels is heated by means of the furnace g, the fire being directly under kettle f, while the heat for the other kettles passes through the arches h. The kettle f is thus in contact with the highest temperature.

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In operating the train, the kettles are filled with juice, which is then heated to boiling. As the water evaporates, the juice is ladled from kettle to kettle, to compensate for its decreasing volume, and at the same time the scum that rises to the surface is removed with a skimmer. The kettle *f* is termed the *strike pan*. The sirup is concentrated in this kettle to the point of crystallization and is then ladled into

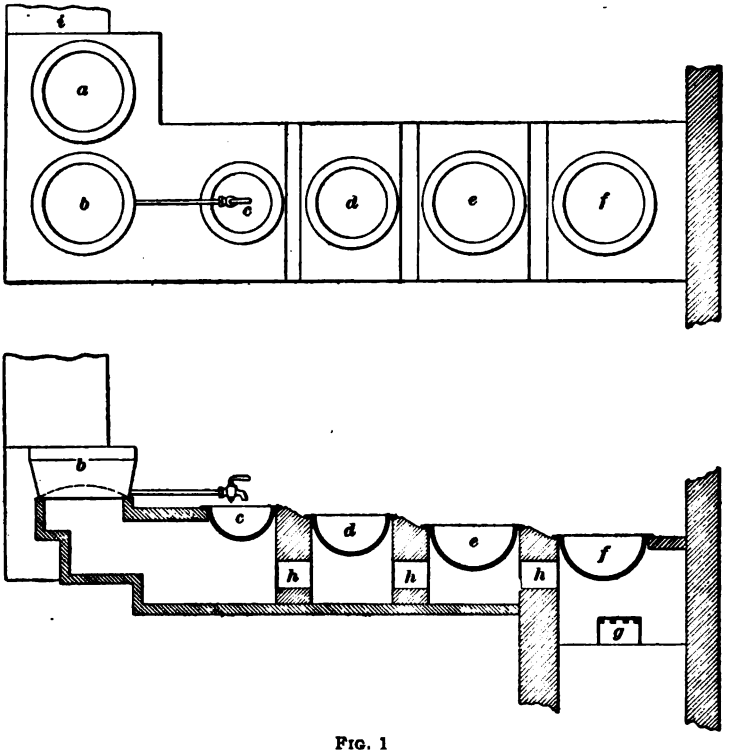


FIG. 1

wooden coolers, where it soon forms a mass of crystals and molasses. In many tropical countries, notably in parts of Mexico and Central America, the evaporation is carried to such a point that the dense sirup solidifies on cooling in suitable wooden molds, forming a very dry concrete that may be wrapped in leaves and shipped in this form for immediate consumption.

3. The steam train differs from the kettles in that steam is used for heating instead of an open fire. The pans of the train are sometimes connected with one another, so that the liquor may flow from vessel to vessel. There are several modifications of the steam train. These include evaporators consisting of a single vessel, into which a constant stream of juice flows at one end and heavy sirup is drawn off at the other.

4. Multiple-Effect Evaporation.—All large factories concentrate the liquors in vacuo in **multiple effect.** With multiple-effect evaporators, as the name indicates, the heat applied in boiling the liquor is not lost after one application, but, in the form of the steam generated in this work, is made to evaporate the liquid in a second vessel, and so on through several applications. In order to accomplish this multiple application of the heat, the vessels, or pans, are closed and the air is partly exhausted from them, the vacuum increasing from pan to pan, thus reducing the temperature at which the liquid boils. The steam generated in boiling the liquid in the first vessel passes through a suitable pipe to the heating surface of the second pan, where it is condensed in evaporating the liquid in this vessel, and thus produces a partial vacuum in the first pan of the system. A similar utilization of the vapor generated in the second vessel is applied in the third, and a partial vacuum is produced in the second vessel. The last vessel of the series is connected, through a condenser, with a vacuum pump, which produces a very high vacuum in this pan. The water of condensation is led off from the first pan through a trap, and is removed from the others by one or more pumps. This water is generally utilized in feeding the boilers, so besides furnishing a very pure water for this purpose, it supplies the boilers with water that is partly heated.

It is evident that the economy of fuel with multiple-effect evaporation is very large. Roughly speaking, assuming that fuel equivalent to 1 pound of coal burned under a boiler will evaporate $7\frac{1}{2}$ pounds of water, in a multiple effect composed

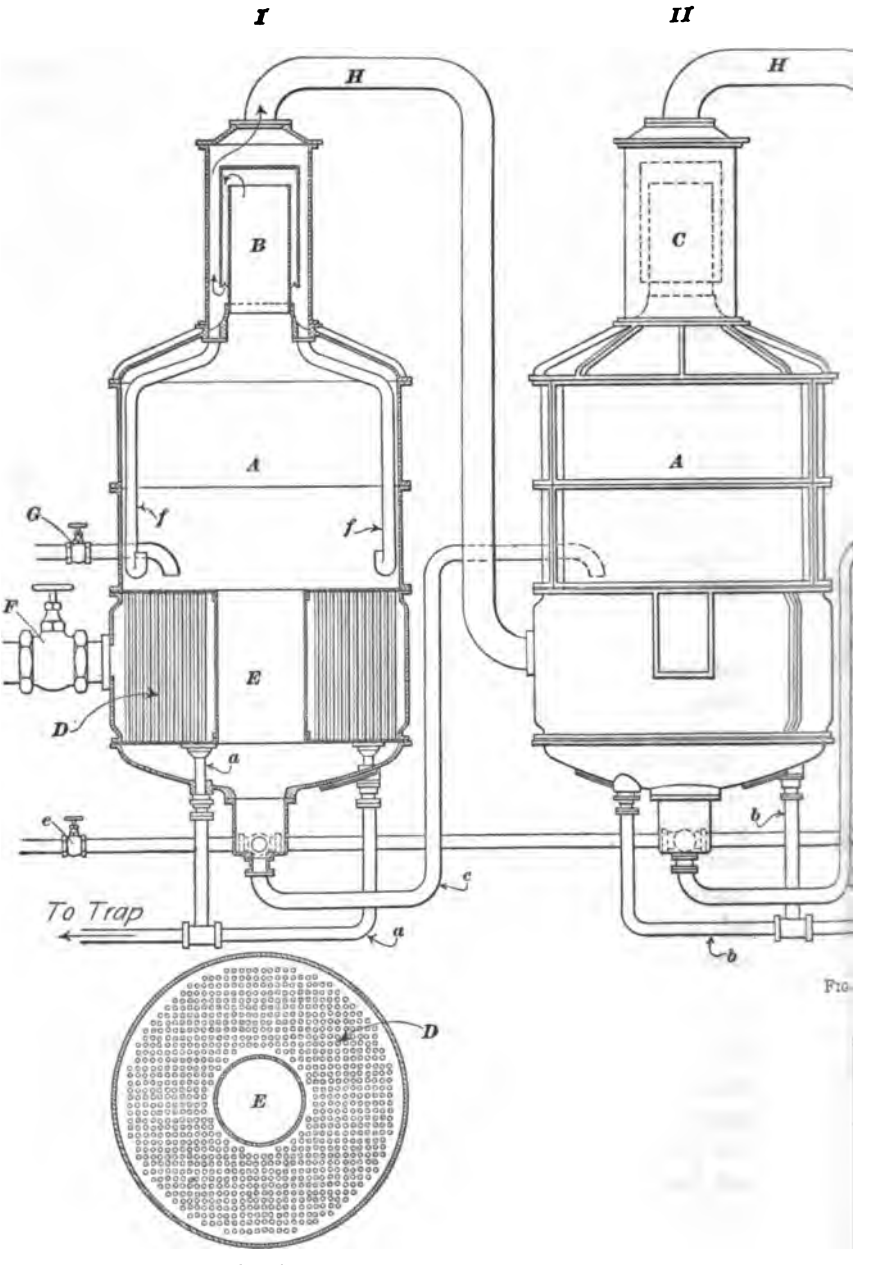
of two vessels, it will evaporate 15 pounds of water, and in a system of three pans, 22½ pounds. These figures are only rough approximations. Exhaust steam from the various pumps and engines of the factory is used for multiple-effect evaporation.

5. Kinds of Multiple Effects.—Multiple effects are usually composed of two, three, or four vessels, and are termed, respectively, *double*, *triple*, or *quadruple effects*. At the present time, double effects are seldom used. The triple effect is usually installed in modern factories, though many large establishments use quadruple effects. A larger number of vessels than four in a series is rarely employed.

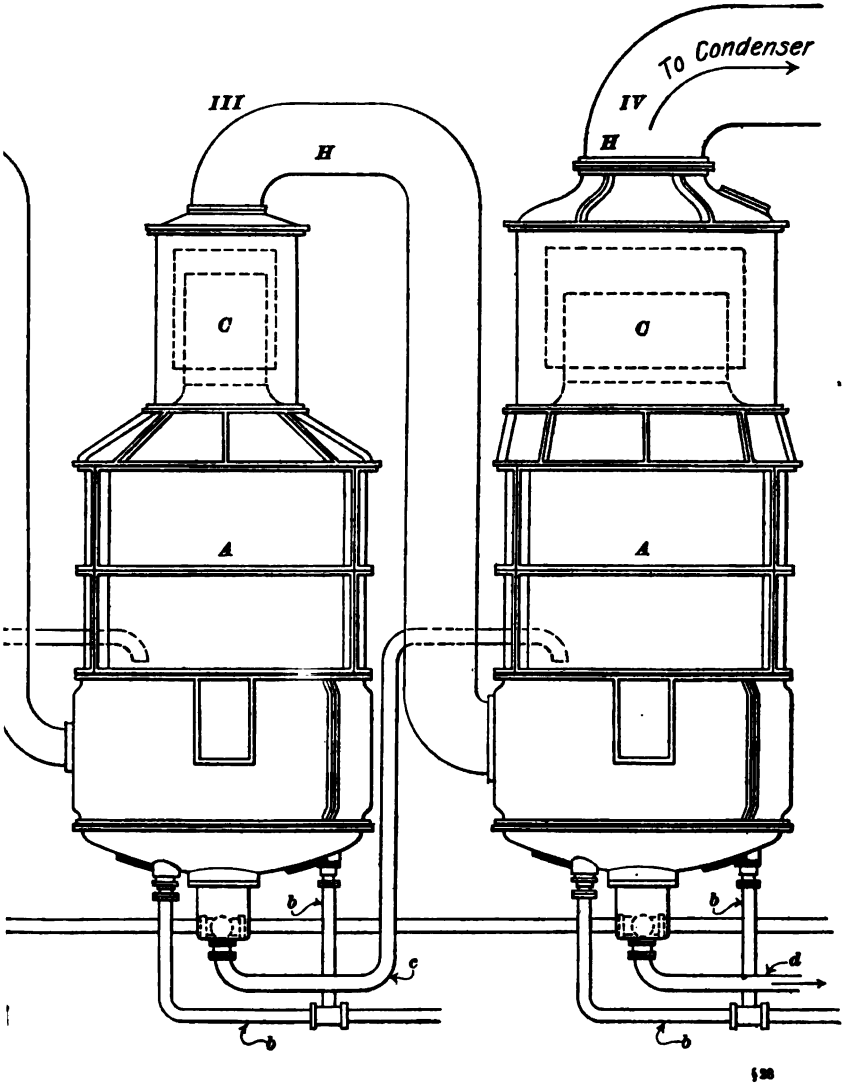
There are many types of multiple effects in use in the sugar and other industries, but the principle on which these are based is the same for all. The form of multiple effect usually employed in the cane-sugar industry is shown in Fig. 2, and is called the **standard type of evaporator.** Since it is very important that the construction and manipulations of a multiple effect should be understood, this apparatus will be described in detail.

6. Quadruple-Effect Evaporator.—The evaporator shown in Fig. 2 is based on the drawings of a large quadruple effect, having 12,500 square feet of heating surface, that was designed and constructed by Samuel Vickess, M. E., New York, for a factory very recently built in Porto Rico. Only the parts of the apparatus essential to an understanding of its construction and manipulation are shown in the illustration. The dimensions are those of the Porto Rico quadruple effect, and are given to illustrate the variations in the construction of certain parts of the vessels that were necessary on account of the differences in vacuum.

Referring to Fig. 2, A, A, A, A are cylindrical cast-iron vessels, or pans, each 9 feet 3 inches in diameter. At D of each vessel there is a drum, or belt, fitted with a large number of brass tubes and a large iron tube E. Each brass tube is 5 feet 1½ inches long by 1½ inches in diameter, and the tube E is 3 feet in diameter. The drum D is called the



Section



120

calandria, and the tube *E*, the *down-take*. A section of the vessel through the calandria shows the arrangement of brass heating tubes *D*, and the down-take *E*.

7. When the apparatus is in operation, fine particles of juice are projected from the boiling liquor and are carried upwards with the vapors. This is termed *entrainment*. To avoid the loss of this juice, save-alls are located in the vapor pipes at *B*, *C*, *C*, *C*. The vapors follow the path in the save-all indicated by the arrows. As shown at *B*, a hood with serrations on its lower edges fits over the neck of the vessel. The particles of liquid entrained with the vapors strike against the walls of the hood and are held there until they drip to its lower edge. Here, the serrations gather them into large drops, which fall into a suitable canal and are led back into the vessel through the pipes *f*. These pipes are curved at the lower ends, so as to trap a portion of the liquid and thus prevent the vapors from passing through them.

The vessels are connected with one another by pipes *H*, which lead from the vapor space of one to the calandria of the next, with the exception of the vapor pipe of the last pan, which is connected, through a condenser, with a powerful vacuum pump. The types of vacuum pumps will be described later.

8. Since, as just explained, the first vessel of a quadruple effect is usually operated under low pressure and the others with a vacuum, which increases from pan to pan, the volumes of the vapors from equal quantities of boiling liquor also increase from pan to pan, being greater the higher the vacuum. Provision for this increase in volume is made by increasing the size of the save-alls *C* and the vapor pipes *H*. The diameters of the necks of the vessels at the save-alls *B*, *C* in the Porto Rico apparatus are, respectively, 2 feet, 2 feet 2 inches, 2 feet 9 inches, and 5 feet 6 inches, and the corresponding dimensions of the vapor pipes *H* of the first three effects are 18 inches, 20 inches, and 46 inches. If it were not for this enlargement of these parts of the apparatus, the vapors would have to travel at a very high velocity, the entrainment of juice would be

excessive, and the vapors could not escape with sufficient rapidity.

9. Aside from the loss of sugar involved, entrainment is very objectionable, since the water obtained from the condensation of the vapors is used in feeding the steam boilers. When the feedwater contains an appreciable quantity of sugar, foaming is produced, and this may endanger the safety of the boiler.

The upper part of the steam space of each calandria is connected by means of a small pipe, which is provided with suitable regulating valves, either with the vapor pipe of the last effect or with its own vapor pipe. The purpose of these pipes is to remove the ammoniacal gas that results from the decomposition of certain nitrogenous constituents of the juice. The gas is lighter than the steam; hence it rises to the upper part of the calandria. The quantity of ammoniacal gas produced in evaporating cane juice is very small, but the contrary is the case in treating beet juices. The quantity of the gas in beet-sugar manufacture is so large that where proper provisions for its removal are not made, the tubes of the evaporator may be destroyed.

10. The remaining details of the construction of the quadruple effect will be explained in the following description of the manipulation of the apparatus:

Referring to Fig. 2, the travel of the juice and vapors follows the order of the Roman numerals. To begin work with the apparatus empty, the vacuum pump is started and juice is admitted into vessel *I* through pipe *G*, into *II* through pipe *c*, and so on. The pipes *G* and *c* are provided with valves so as to regulate the flow of the juice. Having filled each pan with juice to a point a little below the tops of tubes, steam is turned into the calandria of the first pan through the valve *F*. The steam used for this purpose is that exhausted by the various engines of the factory. Sufficient pressure is secured by the use of a back-pressure valve, and deficiencies in the supply of exhaust steam are made up with live steam. The juice in the first pan soon begins to boil,

and the vapor generated passes through the vapor pipe and into the calandria of the second pan, where it is condensed in boiling the liquor in this vessel; in a like manner, the juice is boiled in the third and fourth pans. The vacuum in the last pan is produced by the condensation of the vapors from that vessel and the assistance of the vacuum pump; that in the second and third pans, by the condensation of their vapors in the calandrias of the third and fourth pans, respectively; and in the first, or pressure, pan, as it is often termed, the pressure or vacuum maintained is regulated by the amount of steam admitted at the valve *F*.

The condensation water from the calandria of vessel *I* flows through the pipes *a* and a steam trap to the feedwater tank of the boilers; that from the remaining pans is removed by means of pumps.

11. The evaporation having been started as described, the level of the boiling liquor in each vessel should be maintained high enough to just cover the tubes of the calandria. This is done by adjusting the flow of juice through the pipes *G* and *c* by means of the valves at each pan. The sirup is pumped from the last pan through the pipe *d* into the storage tanks. The flow of sirup to the pump is regulated by a valve in pipe *d*. This valve is opened or closed a little at a time until sirup of the desired density is obtained. It should be noted that a current of juice flows through the apparatus from pan to pan, fresh juice entering at the pipe *G* and finished sirup being pumped out of the last vessel at *d*. The process is therefore a continuous one.

The steam pressure in the calandria of vessel *I* is kept as low as is consistent with obtaining sufficient duty from the apparatus and maintaining a regular flow of liquor from pan to pan. Under ordinary working conditions, the gauges connected with the vapor spaces of the vessels will show the following indications: *I*, 2 to 3 inches (mercury) pressure; *II*, 5 to 7 inches vacuum; *III*, 15 to 16 inches vacuum; *IV*, 27+ inches vacuum. The vacuum in the fourth pan should be as high as the condenser and pump will produce. The

pressure in the first pan and the vacuums in the second and third are regulated by the admission of steam to the calandria of the first pan.

The vacuum conditions are somewhat different in operating a triple effect. In such a case, the gauges show, respectively, 3 to 5 inches vacuum, 15 to 17 inches vacuum, and 27+ inches vacuum.

12. Chapman's Circulators.—In order to promote the circulation of the liquor through the tubes and to increase the capacity of the evaporator, **Chapman's circulators** are frequently used. In installing the circulators, a diaphragm is placed at the bottom of the down-take *E*, Fig. 2, of each vessel, and a tube, forming an inverted siphon, connects the space above the diaphragm of one pan with that below the diaphragm of the next vessel. The pipes *G* and *c* are not used. The space above the diaphragm of the fourth pan is connected directly with the sirup pump. The clarified juice is delivered to the first pan, below the diaphragm, by means of a pipe provided with a regulating valve. Since there is more or less steam formed as the juice passes from pan to pan, due to the difference of vacuum, a separating device is used, which insures the delivery of the juice free from vapor, where required. The height from the top of the calandria to the lowest part of the siphon must be such that the latter will always be filled with liquor, and thus prevent the vapor from checking the flow of juice.

13. The operation of the apparatus may be described as follows: Assuming that the pans have been charged with juice, the liquor boils up through the tubes *D* of vessel *I*, overflows into *E*, and is carried by the siphon to the lower compartment of *II*; it then boils up through the tubes of that pan, overflows as before, and passes through the siphon to pan *III*; and so on. The liquor that overflows into the down-take of vessel *IV* is pumped to the sirup storage tanks. If the density of this sirup is too high, the attendant increases the flow of juice into the first pan, and vice versa. When using the circulators, it is simply necessary to regu-

late the steam pressure in the calandria of the first pan and the quantity of liquor in that pan. If attention is paid to these details, the siphons will maintain the proper level of liquor in the other pans. The rapid circulation of the juice through the tubes of the calandria promotes the evaporation and reduces the fouling of the heating surfaces.

14. Formation of Scale and Its Removal.—All metal surfaces used in heating and evaporating cane juices rapidly foul, or scale. The material that deposits on the heating surfaces and bakes to a scale is a very poor conductor of heat and greatly reduces the efficiency of the apparatus.

TABLE I

COMPOSITION OF SCALE

Ingredients	1st Pan Per Cent.	2d Pan Per Cent.	3d Pan Per Cent.	4th Pan Per Cent.
Phosphate of lime	57.85	56.98	15.02	7.49
Sulphate of lime	2.02	1.92	.54	1.65
Carbonate of lime	3.25	4.68	19.55	9.93
Silicate of lime	7.86	13.31	.71	7.02
Oxalate of lime			11.32	11.27
Iron oxide	2.03	1.53	2.31	2.58
Silica	7.79	7.43	39.26	54.34
Combustible matter . . .	20.37	13.41	11.04	5.08

The tendency of the juice to form scale varies with the locality in which the cane is grown. In certain cane districts, a multiple effect can be operated only a week before it is necessary to clean its tubes, and in other localities, under the same manufacturing conditions, the heating surfaces scale slowly and the apparatus may be used 2 weeks or more without cleaning. In Table I is given the composition of the scale that formed in a quadruple effect in Java.

15. Comparatively little scale forms in the tubes of the first pan of the multiple effect, but this is not always the

case. The quantity increases from pan to pan as the liquor increases in density, and is always thickest in the last, or sirup, pan.

As will be noted by an inspection of Table I, the composition of the scale differs greatly in the different pans. The scale from the fourth pan, or effect, as the vessels are often styled, would evidently be more readily attacked by caustic soda than by muriatic acid, on account of its large silica and silicate content; this is also true of the scale from the third pan. The first and second effects contain a scale rich in substances that are soluble in muriatic acid, but the scale also contains a large proportion of organic matter. It is apparent from these analyses that boiling caustic-soda solution, followed by muriatic acid, should be very effective in the removal of the scale, and it is with these reagents that the evaporators are usually cleaned.

16. In practice, a strong caustic-soda solution, containing from $1\frac{1}{2}$ to 2 pounds of soda per cubic foot of water, is boiled in the pans for 2 or 3 hours. The soda solution is then run into a storage tank, for future use, and the heating surfaces are rinsed with water. The action of the soda softens the scales, but dissolves very little of this material. This soda treatment alone is often sufficient, but, usually, it is also necessary to boil dilute muriatic acid in the pans. Both the soda and the acid should be boiled with the pans open, that is, at atmospheric pressure. The spent acid is run into the sewer. The soda solution may be used many times by decanting it from the mud, which settles to the bottom of the tank, and occasionally adding fresh caustic soda to it.

The heating surfaces may also be freed from scale by means of tube scrapers. The scale should be kept wet with water during the scraping. This method of cleaning, however, requires so much time that it is usually only available when the manufacture is suspended for a period of several hours.

17. Purification of the Sirup.—The sirup as it comes from the vacuum evaporators contains much matter in sus-

pension that has been separated during the concentration and that was soluble in the thin juice. For the storage of the sirup and the removal of this precipitate, the liquor is run into settling tanks, and, after a suitable period of rest, it is decanted into the vacuum-pan charging tanks. The precipitate is disposed of as previously described.

Many sugar makers heat the sirup until it boils, then skim off the impurities that rise to the surface—that is, clarify it, —and finally run the liquor into settling tanks. As has been noted in *Manufacture of Sugar*, Part 1, sucrose is readily inverted by heat alone, but more rapidly in the presence of air. It is thus evident that great care is necessary in the clarification of sirup, since its boiling point in the open air is above 102° C. To avoid loss of sugar when clarification is practised, the heat should be discontinued when the liquid begins to boil. It is doubtful whether this process is advantageous, except in the manufacture of white sugar.

CRYSTALLIZATION OF SUGAR

18. The sirup obtained by the evaporation of the juice usually has a density* of 54.3° Brix at 17½ C.—corresponding to 30° Baumé and 1.257 specific gravity—and contains about 46 per cent. of water. If only the matter of the further concentration of the sirup were involved, it could be readily and economically accomplished in the multiple effect; but the crystallization of the sugar must also be considered. In all modern factories, the sugar is crystallized, or grained, in a single-effect, vacuum evaporator, termed a *vacuum pan*, and this crystallization is effected while the liquor is boiling. This process requires that the sirup used shall be of moderate density, as will be shown later. The vacuum-pan process of crystallizing sugar has resulted in great economy in the manufacture and in the direct production of high-grade sugars.

* In sugar factories, the word *density* is used synonymously with *specific gravity*, and is expressed either in degrees Brix or in degrees Baumé.

The apparatus used and the methods of procedure followed in "boiling" vacuum-pan sugars will now be described.

19. Vacuum Pan.—A vacuum pan, shown in Fig. 3, consists of a cylindrical cast-iron or copper vessel with a conical bottom and dome-shaped top. Copper, however, is not often used in this construction. A vapor pipe *A* leads from the dome to a save-all *B*, and from this point to a vacuum pump. A large valve, called the *strike*, or *foot-valve*, is located at the bottom of the pan at *C*. This valve is used for discharging the *massecuite;* that is, the mass of sugar crystals and molasses. A series of copper coils—usually four or more in number—fitted with suitable valves *D* forms the heating surface. A large opening, or well, similar to the down-take of the multiple effect, is provided at the centers of the coils. This well serves to promote the circulation of the massecuite and to give access to the pan for repairs. The coils are drained of condensation water through tail-pipes and steam traps. A sirup charge pipe *E* enters the pan either near the strike valve or at a higher point, and in the latter case, passes downwards to near the bottom of the vessel. This pipe has branches that connect with the molasses tanks. Eye-glasses *F* for watching the progress of the work, a proof stick *G* for withdrawing samples of the material for examination, a vacuum gauge *H*, a thermometer *I*, a break-vacuum valve *K*, and a steaming-out pipe *L* complete the usual equipment of the pan.

20. Many factories have a central condenser with which all the pans and multiple effects are connected. With this type of installation, a head-valve is placed in the vapor pipe, to be closed when the pan is not in use. A small pipe connects the pan with the vacuum line, for the purpose of establishing the same pressure on both sides of the head-valve when a "strike" of sugar is to be boiled. Pans are also occasionally arranged for the discharge of the massecuite by air pressure and its distribution among the crystallizers through large pipes. This arrangement also requires a head-valve in the vapor pipe.

21. There are two types of pumps employed to produce the vacuum, namely, that of the wet system and that of the dry system. In the wet system, the water of condensation from the vapors and the condensing water are passed through the pump. In the dry system, the condensation and condensing waters are led off through a Torricellian tube, or "leg pipe," the lower end of which dips below the surface of water into a small tank, called the *hot-well*. The water in this well seals the pipe and at the same time permits the escape of water from the condenser. A small pipe connects the condenser with the vacuum pump, which removes the air from the pan when beginning an operation, or strike, and also the air that enters the apparatus by leakage or that which is entrained by the sirup.

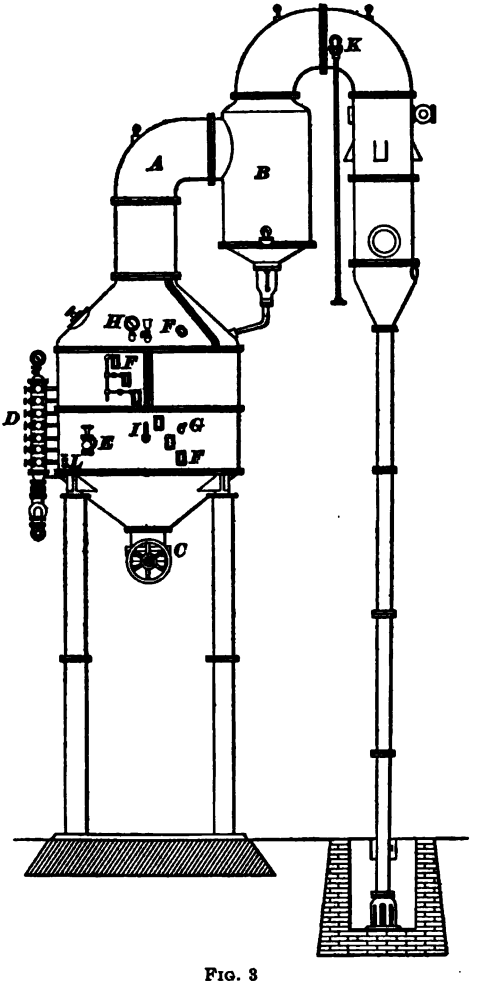


FIG. 3

The degree of exhaustion of the pan, or, to use the usual sugar-house expression, the *vacuum*, is increased or decreased by increasing or decreasing the quantity of water injected

into the condenser. This regulation of the vacuum is of importance, since the temperature of the massecuite must be varied at different stages of the operation of boiling the sugar. When a common condenser is used for several pans, the vacuum is regulated by the head-valve, which must be slightly closed when it is desired to reduce the vacuum.

The heating surfaces of the vacuum pans foul, or scale, but not so rapidly as those of the multiple effect. These surfaces may be readily cleaned by boiling a strong caustic-soda solution in the pan.

22. Vacuum-Pan Process of Boiling Sugar. Having described the apparatus used in the boiling of sugar—the vacuum pan—the methods will next be considered. While it is important that the process of sugar boiling and the principles on which it is based be thoroughly understood, nevertheless, to become proficient in this work, actual practice at the vacuum pan is essential. Practice is necessary in order to learn to judge the relative densities of the liquor under different conditions of purity, vacuum, and temperature, from the appearance of the material as it is boiling in the pan, and from the examination of test samples drawn with the proof stick. Practice is also necessary in order to decide on the necessary manipulations from the quantity and appearance of the crystals in the test samples.

Factory terms will be used in the following descriptions of methods, but will first be defined. The essential parts of the vacuum pan have already been described.

TERMS USED IN SUGAR BOILING

23. Sirup.—The clarified juice of the cane evaporated to a density of about 54° Brix. Sirup* of other densities may be used, but that indicated is customary in good practice.

Grain.—The sugar crystals in the massecuite. This word is also used as a verb, to indicate the process of forming the crystals.

*The refiners apply this word to the final molasses.

Proof.—That stage of the process when the liquor has been concentrated to a state of supersaturation with sugar. In boiling strikes of molasses, the word is modified to "string-proof," "hook-proof," etc., according to the density of the material.

Boiling to Grain.—The boiling of a strike in which the sugar is crystallized in the vacuum pan.

Boiling Blank, or to String Proof.—The boiling of a strike in which the crystallization of the sugar is left to a subsequent operation.

Massecuite.—The thick mixture of sugar crystals and the mother liquid; also, where strikes are boiled blank, or to string-proof, the heavy material, ready for the subsequent crystallization of the sugar, is called *massecuite.* The word is also, for convenience, applied to the material in the pan at all stages of the process. The massecuites are numbered serially, according to their origin, beginning with that from sirup, which is called *first massecuite.*

Mixed Massecuite.—A massecuite formed partly of sirup and partly of molasses. The molasses is said to be "boiled in."

Molasses.—The liquid residue left after removing the sugar from the massecuite. The various grades of molasses are numbered to correspond with the massecuites from which they are obtained.

Strike of Sugar, or Massecuite.—The completed massecuite from a boiling is termed a *strike*, and the operation is called "boiling a strike of sugar, or massecuite." The word strike is also applied, in the same way, to molasses.

Cut.—The part of a strike that is left in a pan as a nucleus on which to boil another strike. This word is also used as a verb; in this sense, the English very often use the word *double.* Many sugar makers use the word cut to indicate the portion of the strike that is removed from the pan.

Cut-Over Pipe.—A pipe used to transfer a portion of an unfinished strike from one pan to another, each portion serving as a nucleus on which to boil a cut strike.

Final Molasses.—The liquid residue from which more sugar cannot usually be profitably extracted.

Circulating, or Condensing, Water.—The water injected into the condenser.

METHODS OF SUGAR BOILING

24. Grained Strikes.—Having closed the foot- and break-vacuum valves of the pan, the vacuum pump is started and the circulating water is turned into the condenser. When the gauge indicates a vacuum in the pan of from 15 to 20 inches, the sugar boiler opens the charge valve and draws sirup into the vessel until the copper coils are covered, or until, from previous experience, he judges that the pan contains sufficient liquid. He now carefully and slowly opens the steam valves, one at a time, beginning with the lowest, or No. 1, as it is called, allowing time for the water of condensation to escape from the coil. Care is necessary, so as to avoid injuring a coil. The sirup soon boils and the water evaporates rapidly from it. When the surface of the liquor falls to near the top coil, the steam valve on that coil must be closed, and so on from coil to coil, never leaving one uncovered while its steam valve is open.

Soon, from the appearance of the sirup and from the flow of the drops projected on the eye glasses, the sugar boiler notes that the liquor is becoming very dense. By gradually closing the circulating-water valve, he raises the temperature of the boiling liquid to about 65° C., or to such other temperature as the grade of sugar he is boiling requires, and by manipulating the water valve maintains this condition. From time to time, a test sample is drawn with the proof stick and is examined on a piece of plain glass by means of transmitted light. In a short time, the boiling liquid becomes saturated and then supersaturated with sugar, at the pan temperature, and microscopic crystals of sugar begin to form.

It requires experience on the part of the pan boiler to detect promptly these minute crystals, which an inexperienced eye would scarcely note. The sugar boiler now carefully

follows the formation of the crystals, and the moment he judges that these are numerically sufficient, he opens the charge valve and draws a small quantity of sirup into the pan; he also slightly closes the circulating-water valve so as to prevent a fall in the temperature of the boiling liquor. A large charge of sirup would cool the boiling mass and thus cause additional crystals to form, and the same thing would occur if the vacuum should rise and thus cool the liquor. The reason for this is that when a solution is saturated with sugar at a certain temperature, it is supersaturated at a lower temperature and the crystals separate.

25. As the charge of sirup evaporates, the panman carefully watches the liquid until it is again saturated, but he avoids supersaturating it by injecting another charge of sirup into the pan. By this procedure, he forces a part of the sugar in the sirup to deposit on the crystals already present, instead of forming new ones, or *false grain*, as this condition is termed. The sugar boiler frequently examines test samples on the piece of glass, and searches for false grain, which he must immediately get rid of if produced. This is accomplished by increasing the temperature of the pan and charging copiously with sirup. The moment the disappearance of the false grain is noted, the panman begins to cool the massecuite gradually, and when the temperature falls to about 65° C., he continues the boiling as before. False grain is objectionable, because it impedes and often obstructs the centrifugating of the sugar. A loss of sugar also occurs by the fine crystals passing through the centrifugal sieves.

The alternate charging and evaporating are continued until the pan is filled with massecuite. The charges of sirup are gradually increased in volume until the pan is about two-thirds full, when they are decreased. During the latter stage of the operation, the massecuite is gradually increased in density from charge to charge, and finally it is evaporated to a water content of about 7 per cent. During this process, the crystals increase from a minute size to that of commercial sugar.

The steam is now shut off from the coils, the vacuum pump is stopped, and the break-vacuum valve is opened. When the vacuum falls to zero, as indicated by the gauge, the massecuite is discharged from the pan through the foot-valve. The further treatment of the massecuite is described in the paragraphs devoted to crystallization in motion and to curing the sugar.

After boiling and discharging a strike, the pan must be thoroughly cleaned by steaming, to recover the last adhering portions of massecuite. This steaming is necessary not only to recover the sugar, but also to prevent it from caramelizing on the coils.

26. At the beginning of the strike, it is important that the pan boiler form a sufficient number of crystals and that he at all times avoid excessively large charges of sirup, otherwise false grain may be formed.

The grade of sugar required and other considerations determine the point in the pan at which the strike is grained. When the crystals are formed between the first and second coils, the strike is said to be *grained low*, and when formed near the middle of the pan, to be *grained high*. Low graining produces comparatively few crystals of large size, and requires long boiling. High graining, on the contrary, produces a fine-grained sugar, with short boiling. When a strike is to be cut over from one pan to another, or an ordinary cut is to be made, except in the production of large crystals, it is usually grained high.

The conduct of the work is necessarily modified somewhat by the use of low-purity sirups or by the boiling in of molasses. With this class of materials, the strike is usually boiled at a high temperature and is "kept close," that is, very dry. When boiling very rich sirups, it is necessary that the strike be "kept very free," or of moderate density.

27. The temperature conditions vary in boiling different grades of sugar. A soft, low-test sugar is produced by boiling the strike at as low a temperature as possible, and clean, hard crystals are obtained by hot boiling. Large crystals

are more readily obtained with thin sirup and the consequent slow evaporation. The commercial granulated sugar is boiled at a high temperature when produced in the factory, and usually also when made in the refineries.

The large-grained sugar of a light-yellow color, known as *Demerara crystals*, is colored in the vacuum pan in the boiling process by means of dilute sulphuric acid. This sugar is made in Demerara, for the British market. The large crystals are usually obtained by "cutting," or "doubling." This is a wasteful process, however, because the acid inverts much of the sucrose of the sirup. Muriate of tin (stannous chloride) is used both in the vacuum pan and in the sugar wash in the centrifugals to obtain a bright or a yellow product.

28. The methods of graining and charging that have been described are not the only ones used. Many sugar boilers, in graining, concentrate the sirup to a certain density and then increase the vacuum, thus cooling the dense sirup until it is supersaturated with sugar and causing the crystals to separate. A small charge of sirup is drawn into the pan at the time of lowering the temperature. This operation is repeated until sufficient crystals are obtained. With many, but not with all installations, the charging may be continuous instead of intermittent. In this method, the charge valve is opened immediately after graining, and from then until the end of the strike, its opening is so regulated from time to time as to maintain a saturated sugar solution.

29. Cut Strikes.—When a **cut strike** is to be boiled, a part of the massecuite from a previous strike is left in the pan, and its crystals take the place of the grain that would otherwise have to be formed. Sufficient massecuite must form the cut so as to supply enough crystal surface for the deposition of the sugar in the sirup, otherwise false grain will result; or, if this is avoided, the molasses surrounding the crystals will not be satisfactorily exhausted of sucrose. This method produces a very large-grained sugar.

30. Blank Strikes, or Crystallization at Rest.—In sugar boiling, the terms *string strikes*, *smooth strikes*, and *jellied strikes* have practically the same meaning as **blank strikes.** Until a few years ago, the only grained sugar boiled was that of the first crystallization, leaving the molasses to be boiled blank. In boiling blank strikes, the molasses is first heated so as to melt the fine crystals it usually contains; it is then drawn into the vacuum pan and is concentrated under as high a vacuum as can be obtained with the apparatus. The density to which the massecuite is concentrated depends largely on the richness of the molasses. The evaporation is usually stopped when a test sample held between the thumb and finger produces a strong thread when these digits are separated. The test is often made by pouring the test sample from the proof stick and noting the appearance of the *string* when it breaks. The temperature of the massecuite at this stage should be as low as possible, and, with an efficient installation and good molasses, is usually about 68° C.

31. Considerable skill is required in boiling blank strikes, since the panman must properly judge the quality of the material largely from its appearance, and must apply the crude test described under varying conditions of purity and temperature. The strikes boiled to a "string-proof" are usually run into small, rectangular sugar wagons or into large tanks to accomplish the crystallization. The wagons of massecuite are placed in a hot room and are allowed to remain there a week or longer at a temperature of about 38° C., this time depending on the purity of the molasses. This method may be termed *crystallization at rest* to distinguish it from the more modern method of *crystallization in motion*, which will be described further on.

Massecuites boiled blank and grain strikes of low purity often foam in the wagons, or tanks. This condition is probably due to a decomposition of the compounds formed during the processes of manufacture. Sugar boilers usually attribute the foaming to the boiling of the massecuite at high

temperatures. When foaming occurs, water should be poured on the surface of the massecuite and the foam then beaten down with a paddle.

32. The products made from blank strikes are often termed *molasses sugars*. These sugars have a low polariscopic test, or polarization, which usually ranges from 80° to 90°. Two grades of molasses sugar are usually made, namely, *seconds* and *thirds*. The third sugar is boiled at the close of one grinding season and left until the beginning of the next, or for a period of 2 or 3 months, before centrifugating.

33. Crystallization in Motion.—The process of **crystallization in motion** was devised in the beet-sugar industry by Wulf, in 1884, and was made a practical process by Stammer and Bock, and it is now extending rapidly in cane-sugar manufacture. While the process possibly does not secure a larger yield of sucrose in the sugars than does the ordinary process, namely, crystallization at rest, it produces as much in a single grade of high-test sugar.

Crystallizers will be described fully in connection with beet-sugar manufacture. For the present purpose, a brief description of the apparatus will suffice. The usual type of crystallizer is a horizontal iron cylinder that has both ends closed and is provided with a charging door, a discharging valve, and a spiral mixer. The cylinder is often fitted with a water-jacket for regulating the temperature of the massecuite.

34. There are several methods of using the crystallizers, the simplest of which is to run a strike, boiled blank, into the apparatus and to mix it, by means of the spiral, for several days, or until there is no further crystallization of sugar. This method produces low-grade sugar and is seldom used.

35. In the usual method, a strike of first sugar is boiled to grain, sufficient diluted molasses being used to so lower its coefficient of purity that it will yield a molasses, on

purging, having a purity of about 55°. This massecuite is usually either centrifugated immediately on leaving the vacuum pan or kept in motion in the crystallizer for several hours. A mixed strike of massecuite is next boiled, using grain formed from sirup or a small quantity of first massecuite as a nucleus, or footing, for the strike. The 55° purity molasses from the previous and other strikes is diluted to 30° Baumé and is boiled in on this footing to form a massecuite of about 73° purity. This massecuite is run into a warm crystallizer and is slowly stirred for from 3 to 4 days, or until the molasses from a sample of it, purged in a laboratory centrifugal, has an apparent coefficient of purity of about 47° or somewhat lower. The massecuite is then centrifugated, using a little wash on the sugar if necessary. A third strike is boiled, using sirup or first massecuite, as before, for a nucleus on which to build the crystals, and the 47° purity molasses is diluted and boiled in so as to form a massecuite of about 60° purity. This massecuite is also run into a warm crystallizer, and is kept in motion as long as is practicable, or until the analysis of test samples shows that the molasses is no longer decreasing in purity. The sugar is then purged.

The temperature of the massecuite in all cases is permitted to fall gradually, often to nearly that of the factory. As the massecuite cools, the sugar is forced out of solution, and owing to the circulation of the crystals through the mass, it deposits itself on these, instead of forming new ones. It is sometimes necessary to dilute the massecuite in the crystallizer a day or more before purging, in order to facilitate the separation of the sugar. At times, this dilution is also advisable when the massecuite has been evaporated until very dry, and the molasses should be added for this purpose after a fall in temperature of a few degrees, otherwise false grain may form. The object of warming the crystallizer before use is to prevent the formation of crystals when the massecuite comes in contact with the metal.

The molasses used for boiling in or for diluting should be heated to free it from crystals of sugar, and for the former

purpose should be used at a temperature about the same as that of the massecuite in the pan.

36. Another method of using a crystallizer is to boil a strike of grained sugar in the usual manner, and having reduced the massecuite to a very low water content, to draw into the pan sufficient hot molasses of about 48° Baumé, little by little, to reduce the massecuite to the desired coefficient of purity. This mixture is now run into a crystallizer and is stirred as usual.

Numerous slight modifications of these methods are used, but those described will answer the present purpose (see also *Manufacture of Sugar*, Part 3). These descriptions refer to raw-sugar manufacture. With Louisiana cane, the work may be conducted with somewhat lower purities of massecuites and molasses than those stated, which apply in Cuba. The tropical-cane molasses of low purity is exceedingly viscous and has a glucose coefficient of about 50, whereas this coefficient often passes 100 in Louisiana molasses. Possibly this condition accounts for the low molasses purities readily obtained in Louisiana.

The process of crystallization in motion not only enables the manufacturer to produce high-test sugars, but also to do so promptly and with little labor. Crystallization at rest necessitates a very large installation of tanks or cars. With this process, rich massecuites require from 7 to 10 days, and the very low grades from 1 to 3 months, for the crystallization of the sugar, as compared with from 3 to 5 days for all grades where crystallization in motion is used.

CURING THE SUGAR

37. Separating Crystallized Sugar From Molasses. The crystals of sugar must be separated from the molasses that surrounds them either by simple drainage or by an application of centrifugal force. The removal of the molasses is termed *purging*, and where centrifugal machines are used it is often called *centrifugating*. In the old open-kettle

factories, the room devoted to the drainage of the sugar was called the *purgery*.

All factories formerly purged the sugar by simple drainage. In this method, the massecuite is packed in hogsheads having perforated bottoms, over which cane leaves or other loose material is placed. Sugar canes are also inserted in the massecuite so as to lead the molasses to the bottoms of the hogsheads. The molasses drains into cisterns over which the hogsheads are placed. The sugar obtained by this method in open-kettle factories is called **muscovado.** The molasses is of very good flavor, and in the United States is called **Louisiana,** or **open-kettle, molasses.** Only small quantities of muscovado sugar and open-kettle molasses are now produced.

In Java, simple drainage is used to remove the molasses from very low-grade sugar having fine crystals. The massecuite is packed in bags, or mats, through which the molasses drains. The resulting sugar contains much molasses and is called *sack sugar.*

38. Centrifugals.—Sugars are separated from the molasses in the modern factories by means of machines termed **centrifugals.** The centrifugal is essentially a perforated metal basket, lined with metallic gauze, and is so arranged that it may be charged with massecuite and then be rapidly revolved. The massecuite distributes itself on the lining, owing to the centrifugal force, and its molasses passes through the perforations, leaving the crystals in the machine.

39. Types of Centrifugals.—Centrifugals are of two types, namely, the *Weston*, or *suspended*, *machine*, and those centrifugals in which the lower end of the vertical shaft, or spindle, is supported by a bearing. The Hepworth machine is an example of the latter type. The Weston and Hepworth types of centrifugals are most generally used in the cane-sugar industry. The baskets are usually made 30, 36, and 40 inches in diameter.

40. Operation of Centrifugals.—The operation of a centrifugal will be described so that the production of the various grades of sugars may be more clearly understood. The basket of the machine is charged with massecuite either while it is at rest or while it is in motion, according to the condition of this material or the preference of the workman. The quantity of massecuite forming a charge depends on the depth of the ring at the top of the basket and also on the quality of the material. With a free-purging massecuite, the charge should be large enough to form a wall, when the machine is in motion, the depth of the ring in thickness. With gummy massecuites, this thickness must be greatly reduced. Having charged the machine, its speed is quickly increased to that for which it is designed. The speed varies with the diameter of the basket. A 30-inch basket is usually driven at the rate of 1,200 revolutions per minute, while the larger sizes are driven at a lower number, but at the same periphery speed. As the speed increases, the molasses leaves the crystals, passes through the perforations of the sieve, or lining, strikes the outer casing of the machine, and then drips into a gutter.

41. If **unwashed raw sugar** is to be made, the workman stops the machine and discharges the sugar from the basket through an opening at the bottom just as soon as he notes that no more molasses is being thrown off. In using certain European makes of centrifugals, the sugar is lifted from the basket by means of a paddle. This sugar is almost free from molasses and is ready to be packed for shipment.

If the sugar that is dried as just described does not test high enough with the polariscope, its polarization may be increased by washing it while it is still revolving in the machine. When the sugar is to be washed, the workman watches for the disappearance of the molasses from the face of the wall, and then sprays a small quantity of water or other wash on it from a special form of syringe or some other device. The water softens the molasses and washes it from the surfaces of the crystals.

42. In making **white sugars,** they are washed as just described, except that large quantities of water and a blue wash are used. The blue wash, usually prepared with ultramarine, is for the purpose of "killing" the slight yellowish tinge of color that usually persists in white sugars made directly from the cane.

43. Yellow sugars are brightened, or their color is heightened if need be, by a wash containing muriate of tin (stannous chloride). Even if the color of the sugar at the time of purging is satisfactory without the tin wash, it would not long remain so. The crystals would soon lose their brightness. The quantity of the tin salt that adheres to the sugar is an extremely faint trace, but it is sufficient to insure the keeping qualities of the product.

44. Granulation of Sugar.—White sugar is often submitted to a drying or granulating process after it leaves the centrifugal. This drying apparatus is termed a *granulator*, and the product is the **granulated sugar** of the markets. The granulator not only dries the crystals, but, as its name implies, separates them from one another.

This apparatus is a long iron cylinder about 5 feet or more in diameter, and is so arranged that it may be slowly revolved in a slightly inclined position. Angle irons, forming shallow shelves, are attached to the inner surface of the cylinder at short distances from one another and extend lengthwise of it. An exhaust fan is located at the sugar inlet end of the apparatus, and serves to draw a current of air through a steam heater and thence through the granulator. The ends of the apparatus are boxed in so as to direct the air to the fan.

45. When the granulator is in operation, an elevator delivers an almost continuous stream of moist sugar through a chute into the higher end of the revolving cylinder, and the shelves pick up the sugar and carry it upwards until it slips from them and falls in a shower through the current of hot air. This operation is repeated until the sugar leaves the cylinder at its lower end, owing to the inclined position

of the apparatus. In its travel through the granulator, the hot air abstracts all the moisture from the sugar, and the motion of the latter separates the crystals from one another. On leaving the granulator the sugar is sifted, to remove lumps, and, in the refineries, to separate it into the *fine*, *standard*, and *coarse granulated sugars* of the markets.

There are several types of granulators, but as the principle on which they are based is the same for all, one of the simplest has been described.

46. The moist sugar may be pressed into molds, so as to form large loaves when subsequently dried. It may also be moistened with white sirup and then pressed into cubes by means of a special machine. The cubes are dried in an oven before packing.

The residual molasses is generally utilized in the manufacture of alcohol or in feeding the draft animals on the plantation.

MANUFACTURE OF SUGAR

(PART 3)

BEET SUGAR

INTRODUCTION

1. The manufacture of beet sugar has been practiced in Europe for more than a hundred years; in fact nearly all the sugar consumed there is obtained from this source. The beet-sugar industry in the United States, however, is of very recent date. The sugar produced prior to 1889 was derived almost exclusively from the tropical cane in Louisiana. The refining branch of the industry depended on foreign sources for nearly its entire supply of raw sugars, which was obtained from both cane- and beet-sugar producing countries.

The work of E. H. Dyer, in a small, poorly equipped factory, demonstrated that an American beet-sugar industry could be developed. E. C. Burr, H. T. Oxnard, Claus Spreckles, and others, following Dyer, developed this industry until now it is a large element in the domestic sugar production. The United States Department of Agriculture, first through the efforts of Dr. William McMurtrie and later through those of Dr. H. W. Wiley, has played an important part in developing the beet-sugar industry.

2. Climatic Conditions.—The most favorable climatic conditions for the sugar beet are found where the mean summer temperature is 70° F. Dr. William McMurtrie

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traced a mean isotherm of 70° F., based on observations made during 10 years for the months of June, July, and August, and considered the beet area to be in the vicinity of this line, where the minimum rainfall for the three summer months is 2 inches per month. At the instance of Dr. H. W. Wiley, this isotherm was retraced, and from this and other data he concluded that the American sugar-beet area is within a belt extending 100 miles each side of the 70° F. isotherm. Doubtless, there are many localities near this belt where the conditions are favorable to the beet.

The sugar beet requires an abundance of sunshine for its proper development as a source of sugar; hence, northern latitudes usually produce the richest beets. The plant grows luxuriantly in the South, but the roots rarely contain paying quantities of sugar. Experience has demonstrated that beets of satisfactory richness are not produced very far south of the belt defined by Wiley.

The question of rainfall is also of great importance. Summer rains are very desirable, but heavy autumn rains, especially following a dry summer, are extremely unfavorable to the sugar content of the beet. Late rains cause a second growth of the plant at the expense of the sugar. Cold winters, without alternate freezing and thawing, are desirable for the proper keeping of the roots in storage preparatory to the manufacture.

To recapitulate, Doctor McMurtrie stated the favorable meteorological conditions to be in general, "Comparatively dry and warm spring months during the time for preparation of the soil, planting, and cultivating the crop; moderate temperature, abundant and frequent rains during the summer months * * * ; cool, dry fall, the time for ripening, harvesting, and storing the crop."

3. Varieties of Beets.—The several varieties of sugar beets now known have been derived by selecting roots that possessed valuable sugar-producing qualities. All the varieties are similar to those of the common garden beet, or *Beta vulgaris.*

Among the first, at least in France, to make scientific investigations and experiments for the selection of rich varieties of beets and for fixing these qualities was Louis de Vilmorin, who was ably followed in this work by his son, Henri de Vilmorin. The roots for producing seed were selected for their shape, size, and color, the character of



FIG. 1

their foliage, their keeping qualities, and their high sugar content. Many investigators in various parts of Europe, and recently in the United States, have continued these and other investigations, until there are now many valuable varieties whose characteristics are fixed.

Among the varieties grown are Vilmorin's Improved White beet, French Very Rich, Klein Wanzlebener, White

Imperial, and many others. Many of the very rich beets have been derived from Vilmorin's Improved and the Klein Wanzlebener by systematic selection. Different varieties are chosen by the beet growers for planting under different soil and climatic conditions.

4. Typical Beet.—A typical beet, so far as shape and foliage is concerned, is shown in Fig. 1. This beet is large and tapering, with only a small neck, or crown, and with broad, spreading foliage. These conditions of shape facilitate harvesting. The long tap root penetrates the soil deeply in search of moisture; the spreading leaves shade the adjacent soil, thus conserving the moisture and preventing the growth of weeds; and the small neck, which must be removed in harvesting, results in little waste.

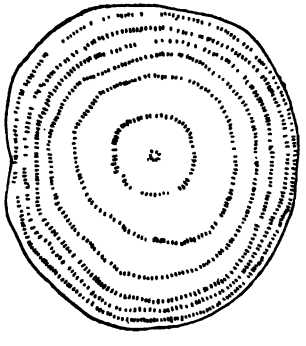


FIG. 2

Beets weighing more than 3 pounds are usually of inferior quality, but this depends more or less on the variety of seed, the suitability of the soil, and the conscientiousness with which the latter has been prepared and cultivated. As a general rule, however, the larger beets are poorer in quality, so far as the sugar content is concerned, than the smaller or medium-sized ones. The beets most eagerly sought by sugar manufacturers are the ones that weigh between 1 and 2 pounds. Where beets are too large or too poor in quality to be used in sugar manufacturing, they must not be considered entirely valueless, as they may be well and advantageously utilized as feed for live stock. Fig. 2 illustrates a cross-section of a beet, the dotted lines showing the concentric rings of growth.

5. Planting.—The beet seed should be planted as early in the spring as the weather conditions will permit. The quantity of seed required per acre varies greatly. The seed germinates more quickly in some soils than in others and

many things unite to act against the germination of every seed, so that it has been found best to plant enough to insure a good stand and also to supply the force necessary to lift the crust of the soil and allow the tender sprouts to reach light and air. It is generally conceded that each acre of land requires from 12 to 20 pounds of seed. The rows should not be less than 18, nor more than 20, inches apart, this space being sufficient for a horse to walk in during cultivation and permitting the leaves of the full-grown plant to shade the ground, thus avoiding rapid drying of the soil. The proper depth for planting depends very much on the locality. Where the atmosphere is humid and the ground moist, from ½ to 1 inch is deep enough to lay the seed, while in very arid regions, 1½ inches is necessary, owing to the rapid evaporation of the surface moisture. Where conditions permit, shallow planting is certainly advantageous.

6. Thinning.—When the seed has sprouted about 2 inches above the ground and the plants show four leaves, it will be found that they stand too close together. It then becomes necessary to thin them so that the remaining ones will be a uniform distance apart. This is done most economically by using a hoe that is 4 inches wide and has a sharp point. Spaces are struck out with the hoe, and small bunches of beets, from 6 to 8 inches apart, are left for two or three "thinners," who deftly pull out all but the healthiest beet. Only one beet is left in a place, since otherwise the roots twist about one another and in the end none of the beets shows a healthy growth or is really worth harvesting. In poor soil, the beets should be thinned until about 10 inches apart; in fairly rich soil, 8 inches is preferable; and in very rich soil, the distance can be shortened to 6 inches. Large beets, as has been stated, are not the most desirable; hence, in thinning the plants, care should be exercised to avoid allowing too much space to each. As a rule, the plant will look weak and sickly after thinning, and generally about 48 hours is required before it recovers sufficient strength to hold up its head again.

7. Cultivation.—The sugar beet is a plant that requires plenty of nourishment, and it is to the interest of the beet raiser to keep the soil as free as possible from weeds. Cultivation should begin soon after the rows are well defined, and should be repeated frequently. The old-fashioned ground cultivator is not suitable for this purpose, but there are numerous beet cultivators to be had. The Moline beet cultivator is considered to be one of the most efficient and is extensively used at present. Whatever cultivator is employed, the soil should be kept mellow and free from weeds. The crown of the beet, from the point where the foliage sprouts, should not be covered by the soil, nor should the beet be exposed too much. Laceration of the leaves should be avoided after they are large enough to protect the exposed part of the beet from the heat of the sun, and from this period until the time of harvesting arrives, it is desirable to keep the field free from weeds by hand. Experience has shown that one man is well able to keep a field of 20 acres clean.

8. Harvesting.—The exact time of maturity of the beets can only be determined by chemical analysis, but experience has demonstrated that when the period of maturity arrives, the dark-green color of the leaves disappears, the outer leaves wither and die, and the center leaves assume a reddish, golden-yellow color. Beets seeded very late in the season often fail to show these characteristic marks of maturity, yet the analysis shows a high percentage of sugar with a corresponding high coefficient of purity. When the chemist discovers that the beet is at its highest sugar content and coefficient of purity—that is, from 14 to 20 per cent. sugar, with a coefficient of from 80 to 85—the crop should be harvested as quickly as possible.

The factory arranges its contracts so that the delivery of beets can be made with regularity, and that all roots may be out of the ground before it freezes so hard as to prevent harvesting. The beets should be harvested before there is danger of a second growth.

When the beets are mature, they are loosened either by means of a strongly built plow or by a special harvester that lifts them. The plow or the harvester breaks the tap root and permits the beet to be readily removed from the soil by means of its leaves. After removing the beet from the ground, it is *topped;* that is, the neck is removed at the lowest leaf scars with a strong knife. The neck, or crown, of the beet contains comparatively little sucrose and a large proportion of mineral matter that is objectionable in the manufacture of sugar. The tops are usually left to rot in the fields, thus returning considerable valuable fertilizing material to the soil. After topping, the beets are thrown into heaps, and if not to be immediately removed to the factory, they are covered with leaves to protect them from the sun.

9. Wide-tired wagons are used to convey the beets from the fields to the cars or storage sheds. There are several dumping devices in use to lessen the labor of unloading of the wagons. Among the simplest of these is a tilting platform on which the wagon is clamped and then inclined to discharge the beets. This method is used where the beets reach the sheds comparatively free from adhering soil.

In certain localities, notably in California, the beets are loaded on a strong net spread over the wagon. When ready to discharge the load, one side of the net is made fast to the car, or bin, and ropes attached to the free side are passed over the car, or bin; then the load of beets is pulled from the wagon by a team of horses. In another method, the loaded net is lifted from a wagon, or car, by a traveling crane and its contents is then deposited where desired in the bin. Hydraulic dumping devices are often used to unload railway cars. Several cars are clamped on a platform and are discharged at one time by tilting them.

About 50 pounds or more of beets is removed from each load, so that the allowance for tare may be estimated. These beets are weighed, then brushed and washed free from soil and rootlets, and parts of the necks that are carelessly left in topping are removed. After cleaning, the beets are dried

by wiping or by drainage and are again weighed. A small arbitrary allowance is often made for the water that is absorbed by the beets or that adheres to them. The loss of weight is used in calculating the deduction to be made for tare. The chemist of the factory usually has charge of the tare room.

10. Storage of Beets.—As the factories are active long after the proper time for harvesting the crop, large quantities of beets must be stored for use during this period. The method of storing depends on local conditions, the climate, and the facilities possessed by the factory.

Covered bins, supplemented by storage in the open yard, serve the purpose in California, where the climate is very mild. The piles of beets should not be very high and they should be protected from the sun by a light covering. Great care is required to avoid loss through the heating of the beets in the bins or the piles. Covered bins are also used in other parts of the United States.

Where beets are to be stored for only a short time, they may be collected in large heaps and then be protected with a light covering of straw. The storage place should always be well drained. In parts of the United States where the winters are cold, the beets are often stored in large piles with no covering. The outer layers freeze, but the frost does not penetrate far into the pile. If exposed to alternate freezing and thawing weather, the outer layers rapidly deteriorate. American experience indicates that in cold climates the beets should be protected from thawing rather than from freezing. When beets are stored for a short time in the fields, they are usually protected with a covering of leaves or soil.

In European practice, sheds and silos are employed, while in some localities, storage in the open air is resorted to. The methods of storage in sheds and in the open air do not differ materially from those followed by the American factories. The silos are of various forms, the usual type being arranged as follows: The beets are piled on a well-

drained piece of ground and are then covered with straw and a layer of earth. Ventilators are located quite near one another, and extend upwards from the bottom of the pile of beets. The silo is surrounded by a suitable drainage ditch.

11. Hydraulic Transport of Beets From Bins, Etc. to the Factory.—The beets are usually conveyed from the sheds, or bins, to the washing machines by a stream of water flowing in a sluiceway. The floors of the bin slope toward the sluice, and the latter is covered with loose planks, on which the roots are piled. As the beets are needed, they are pushed into the sluice by the workmen, and at the factory they are lifted from the sluice either by a wheel provided with buckets or by a screw conveyer.

12. Composition of the Sugar Beet.—The sugar beet usually contains from 3 to 5 per cent. or more of *marc*, or matter that is insoluble in water. As a rule, only two sugars, *sucrose* and *raffinose*, are present in the mature beet, though it may also contain invert sugar, which is always found in the immature root.

The amount of sucrose present varies greatly with the variety of the beet and the soil and climatic conditions. Small beets are usually much richer in sugar than large ones. The average sugar content of beets produced for the American factories will probably approximate 14 per cent. Beets containing as little as 11 per cent. of sucrose are not desired by the factories, and are seldom purchased except at a reduced price.

In addition to sucrose, raffinose, and chlorophyl, the beet contains a large variety of compounds, namely: *Organic acids:* Oxalic, formic, citric, malonic, succinic, aconitic, tricarballicylic, oxicitric, malic, and tartaric acids. *Nitrogenous bodies:* Betaine, asparagine, glutamine, leucine, legumine, tyrosine, xanthine, guanine, hypoxanthine, adenine, and carnine. *Non-nitrogenous bodies:* Lecithine, pectose, and coniferine. *Mineral constituents:* Salts of potassium, sodium, rubidium, vanadium, calcium, magnesium, iron, and manganese.

The bases are combined with hydrochloric, sulphuric, nitric, phosphoric, and silicic acids and the organic acids named in the preceding list.

The beet also contains the non-nitrogenous bodies, lecithine and pectose. The latter is converted into soluble pectine by the pectase, which is also present. Of the sugars in the beet, sucrose, and invert sugar have been described in *Manu-*

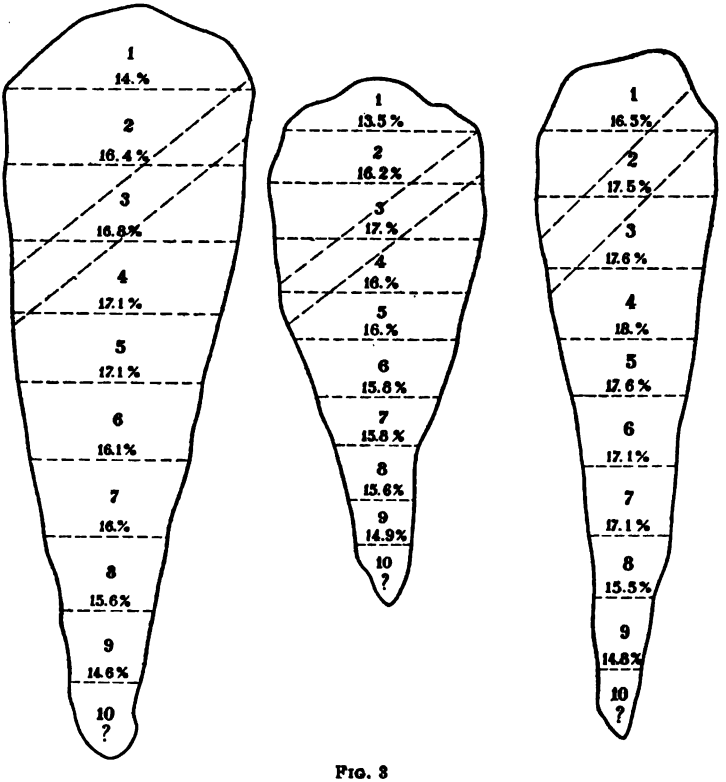


FIG. 3

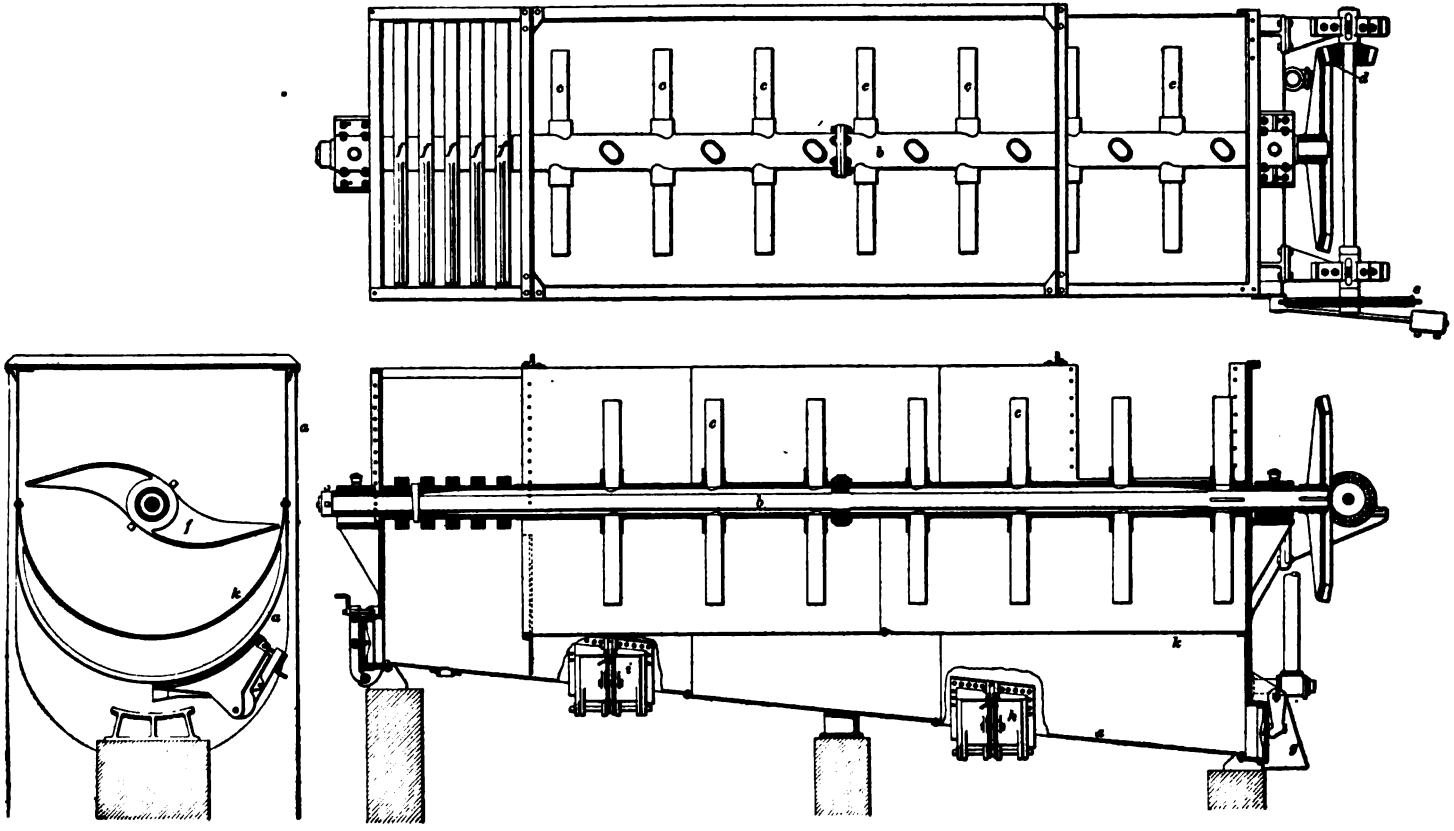
facture of Sugar, Part 1. Raffinose is present in small quantities and accumulates in the molasses. This sugar forms needle-shaped crystals and is not injurious in the manufacture. The distribution of the sugar in the beet, according to Slassky (Bul. Assoc. des Chimistes de France, **12**, 277), is shown in Fig. 3.

13. In the purification of the juice, many of these substances are removed by precipitation as lime compounds. Others are carried down mechanically by the precipitates, and the albuminoids are removed partly by coagulation and partly by precipitation. The precipitant used in the manufacture is lime, the excess of which is converted into the carbonate, which itself acts mechanically in carrying down the suspended impurities of the juice. A part of the lime forms soluble salts, which are troublesome at all subsequent stages of the manufacture. The sodium and potassium salts are not removed in the purification of the beet juice, but remain and give it an alkaline reaction after all the lime has been saturated with carbonic acid.

MANUFACTURE OF BEET SUGAR

SYNOPSIS OF MANUFACTURE

14. The various stages of the manufacture of beet sugar may be outlined as follows: (1) Washing the beets; (2) weighing the beets; (3) slicing the beets; (4) extracting the juice by the diffusion process; (5) straining the juice; (6) purification of the juice; (7) bleaching the juice; (8) concentration of the juice to a sirup; (9) crystallization of the sugar—(*a*) in the vacuum pan and (*b*) supplemental crystallization with motion; (10) curing the sugar; (11) molasses processes, or processes for the recovery of the sugar in the molasses—(*a*) osmose processes and (*b*) saccharate processes; (12) utilization of by-products—(*a*) pulps, (*b*) molasses, and (*c*) distillery and other residues from the utilization of the molasses.



B

FIG. 4

DESCRIPTION OF PROCESSES AND MACHINERY

15. Washing the Beets.—In Fig. 4 is shown a plan view and also a cross-section and a longitudinal section of a **beet washer** made by E. H. Dyer and Company. The washer consists of a shell, within which is a hollow metal shaft *b*, carrying arms *c* that agitate the beets in the water. The beets conveyed from the beds and sheds through a flume containing running water are raised by means of an elevator wheel and are thrown on an apron, which drops them into the washer having a driving gear *d* and *e* at its end. The beets are thrown out of the washer at the opposite side by the arms *f* on to a small conveyer, or into what is called a *trash catcher*, that is, a sheet-metal box with a screw for conveying the beets from the washer to the elevator. This device delivers them to an automatic scale, which registers their weight and drops them directly into the cutter. Referring again to Fig. 4, *g*, *h*, and *i* are doors in the end and sides for cleaning out the dirt, sand, stones, etc. that separate from the beets and accumulate in the space between the perforated false bottom *k* and the shell *a*.

16. Weighing the Beets.—It is customary, especially in Europe, to weigh the beets immediately before slicing them. Preliminary to weighing, the beets are either passed over rotary brushes or transported a short distance on a shaking conveyer, to complete the drainage of the wash water and to remove stones and earth that may still adhere.

Automatic scales are very generally used in Germany and the United States, the Chronos scale, of German manufacture, being usually employed. The filling, weighing, and discharging of this scale are automatic. Numerous semi-automatic weighing devices are also used. The French frequently employ a scale on which the beets are weighed in a car; an automatic stop arrangement prevents the car from leaving the scale until the load is complete.

The automatic weighing of the beets is desirable where the beets are taxed according to their weight.

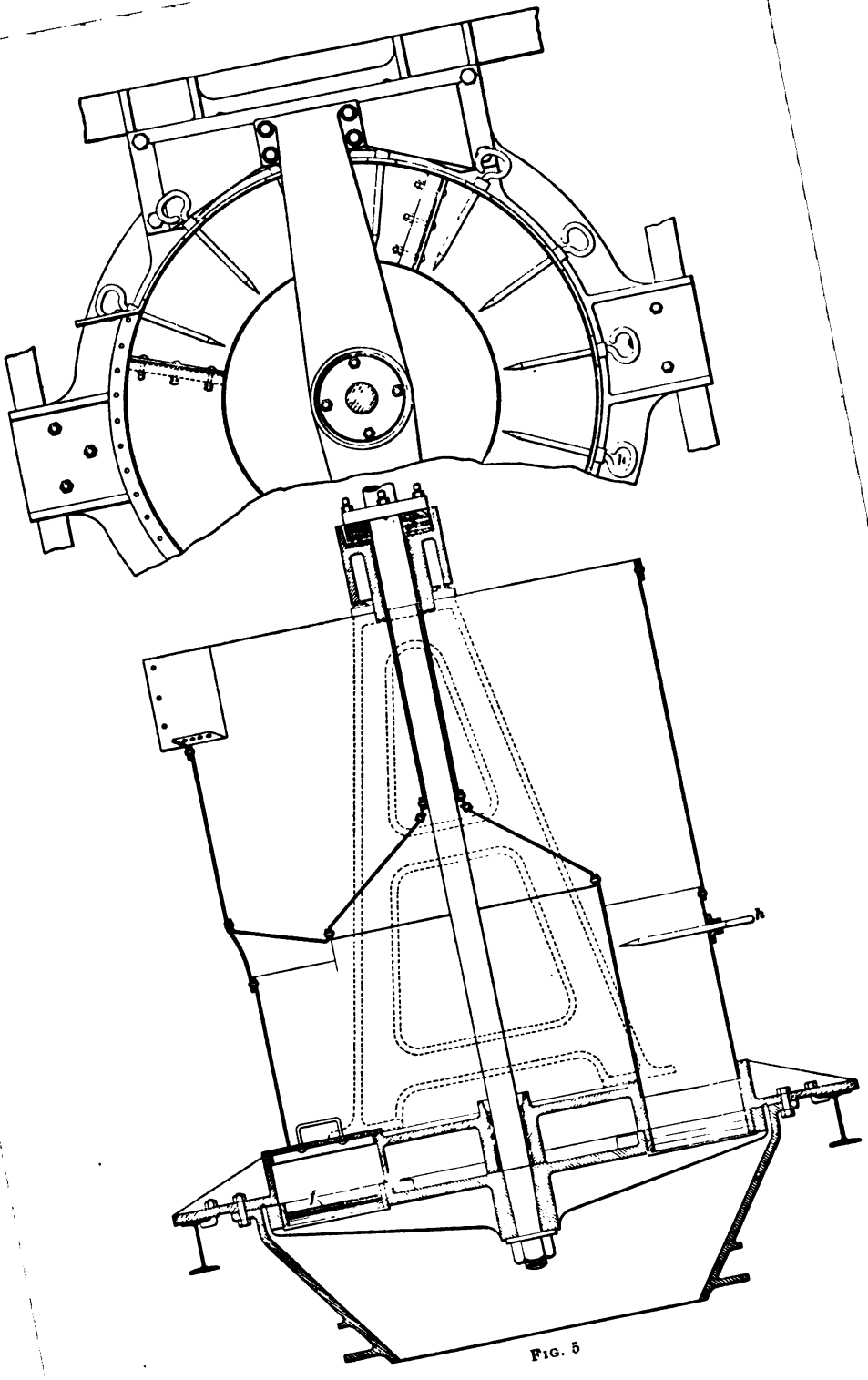


FIG. 5

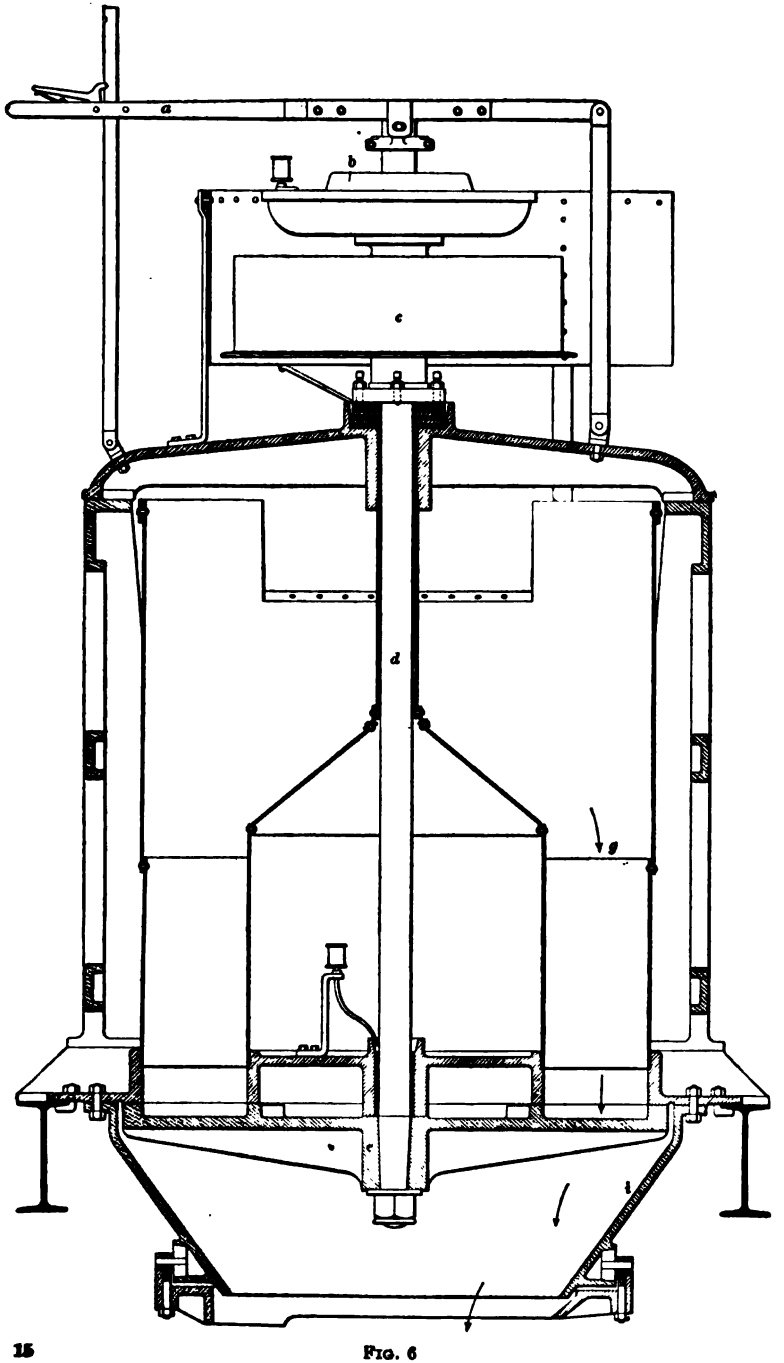


FIG. 6

17. Slicing the Beets.—At the present time, the diffusion process has replaced all others in the extraction of the juice from the beet. The first step in this process, after the beets have been thoroughly cleaned, is to cut the roots into thin slices termed *cossettes*, and it is of great importance that these be of uniform thickness. A machine in common use for this purpose is shown in Figs. 5 and 6. This machine is made by E. H. Dyer and Company.

In the figures, *a* represents the lever for throwing the clutch *b* in and out of action, and *c*, the driving pulley that operates the shaft *d* carrying the circular frame *e*, which holds the knives *f*. At *h* are shown pins used to keep the weight of beets off the knives, so that when dull, they may be replaced by sharp ones without unloading the cutter. The beets fall into the cutter and, following the course of the arrows *g*, are cut by the knives *f* into V-shaped slices, which are usually $\frac{1}{8}$ inch thick and $\frac{5}{8}$ inch wide. These slices, or cossettes, fall through the hopper *i* into a chute, which conveys them to the diffusion-battery cells on the floor below. Some factories use knives that cut a rectangular-shaped cossette, but most of the modern factories prefer the V shape, as in the rectangular shape the walls are much thicker and a higher temperature is required to extract the sugar from them, with the ever-present danger of retarding the circulation of the juice through the cossettes by softening or matting them by excessive heat.

18. Many factories install two cutters for each battery and are thus able to send an uninterrupted stream of cossettes to the battery, as a reserve cutter is always ready for use when the knives of one become dull or broken or in case a cutter for any reason gets out of order. Much trouble and delay are caused by stones, iron, etc. being carried along with the beets and breaking the knives; but this difficulty is partly overcome by *stone catchers* of various designs. The most successful of these are placed in the bottom of the washers, or trash catchers, and are simply sheet-iron boxes kept filled with water, through which the beets flow on their

way to the elevator, the stones falling to the bottom and being removed at regular intervals through a door in the side. Another type consists of a large iron basket with a perforated bottom. The basket is placed under the beet flume, the top of it being on a level with the bottom of the flume. As in the other case, the beets float on over this basket and the stones deposit in it.

EXTRACTION OF THE JUICE

19. Theory of the Diffusion Process.—The juice was formerly extraced from the beet by means of hydraulic presses. This method, however, has been entirely superseded by the **diffusion process.** This process was devised by Jules Robert, in France, and received its first practical application in Austria.

The diffusion process depends, in theory, on the property possessed by a class of substances, called *crystalloids*, of passing from one solution into a contiguous solution, through a porous membrane that separates the two liquids. This action is termed *osmosis*, or *dialysis*. Under the same conditions, certain other bodies, called *colloids*, pass through the membrane, but slowly, if at all. The crystalloids of the beet, of which sugar is the principal one, include most of its mineral salts and salts of the organic acids. Certain albumins are among the colloid bodies of the sugar beet. Crystalloids are substances that may be readily crystallized, and the colloids, on the contrary, are not usually crystallizable. The rate of the dialysis increases as the temperature of the solutions increase.

20. In the application of osmosis, in the extraction of the sugar from the beet, the walls of the plant cells themselves supply the membranes. The beets are cut into very thin slices, and these are then bathed in water. The crystalloids pass through the cell membrane during the interchange of the liquids within and outside the cells.

In slicing the beets, as described previously, many of the plant cells are necessarily ruptured, and their contents mingle

with the water with which the cossettes are bathed in the diffusion process. This process is therefore both one of osmosis and one of maceration. Sharp knives are always used in slicing the beets, so as to avoid rupturing more cells than is actually necessary. The cell membrane consists largely of cellulose and pectin bodies, which are rendered soft and flabby at certain temperatures. The pectin bodies also become somewhat soluble in water on heating; hence, great care must be taken in the diffusion process to have the temperature conditions as near perfect as possible.

It should be observed that ideal conditions cannot be attained in the practical application of the diffusion process and that, together with the crystalloids, many of the colloids are extracted.

21. Diffusion Battery.—The apparatus used in the practical application of the diffusion process consists of a series of vertical iron vessels that are provided with suitable doors for charging them with beet cossettes and discharging the exhausted pulp, and also with piping and connections for the circulation of water, juice, and steam. These vessels may be arranged in a single or a double straight line, or in a circle. The series of vessels is termed a **diffusion battery,** and the individual vessels, *diffusers*, or *cells*.

A conveniently arranged, circular diffusion battery, manufactured by the Walburn-Swenson Company, is shown in Fig. 7. The choice of the type of battery, whether to be arranged in a line or in a circle, depends on the general scheme of the factory and the preferences of the builder. The various arrangements mentioned are equally effective in extracting the sugar. Double-line batteries are usually preferred, as they do not require a very high building and, further, may be readily enlarged to two single-line batteries. The usual number of diffusers forming a battery is twelve.

22. The battery illustrated in Fig. 7 consists of fourteen cells, C_1 to C_{14}, each of which has a feed-opening at the top that may be securely closed with a swinging cover. Each

of these covers has a cock Y, which serves as an air vent and as a means of ascertaining when the diffuser is completely filled with juice. At the bottom of each cell is a hinged door for discharging the exhausted cossettes. This door is opened and closed by means of a hydraulic cylinder K and a balance weight W. Water is supplied the cylinder through the pipe X. Each diffuser has a juice heater, or calorisator, H_1, H_2, etc. and is connected with the bottom of this device by the pipe F (shown in the figure only at cell C_{14}). The upper part of the heater connects through the valve M, and the upper pipe lines connect with the top of the next diffuser of the series. The heaters receive steam through the pipe S and the valves R. Instead of tube or coil heaters, steam injectors are frequently used. The pipes A and B are, respectively, the water and juice lines. The cross-valve N serves to connect the first cell of the series with the water-line A.

In operating the battery, each cell successively becomes the first of the series. Three cells are usually disconnected from the others. One of these three cells is being charged with fresh cossettes, the second is empty and ready to be charged, and the third is being emptied of exhausted pulp. A compressed-air pipe, with valves at each diffuser, is often provided to transfer the water from the cell containing exhausted pulp to the next diffuser, before removing the pulp. In this arrangement, air is used to force the juice through the series of vessels and the juice from the last diffuser into the measuring tank.

23. Diffusion Process.—With the assistance of the outline of the theory of the process and the illustration of the machinery, the following description of the diffusion process itself will be readily understood:

In beginning the work, a diffuser is filled with beet cossettes, and warm water is then passed into it through the pipe at the bottom, while the air and gases are vented by means of the cock in the cover. While the first diffuser is being filled with water, the second is receiving cossettes, and

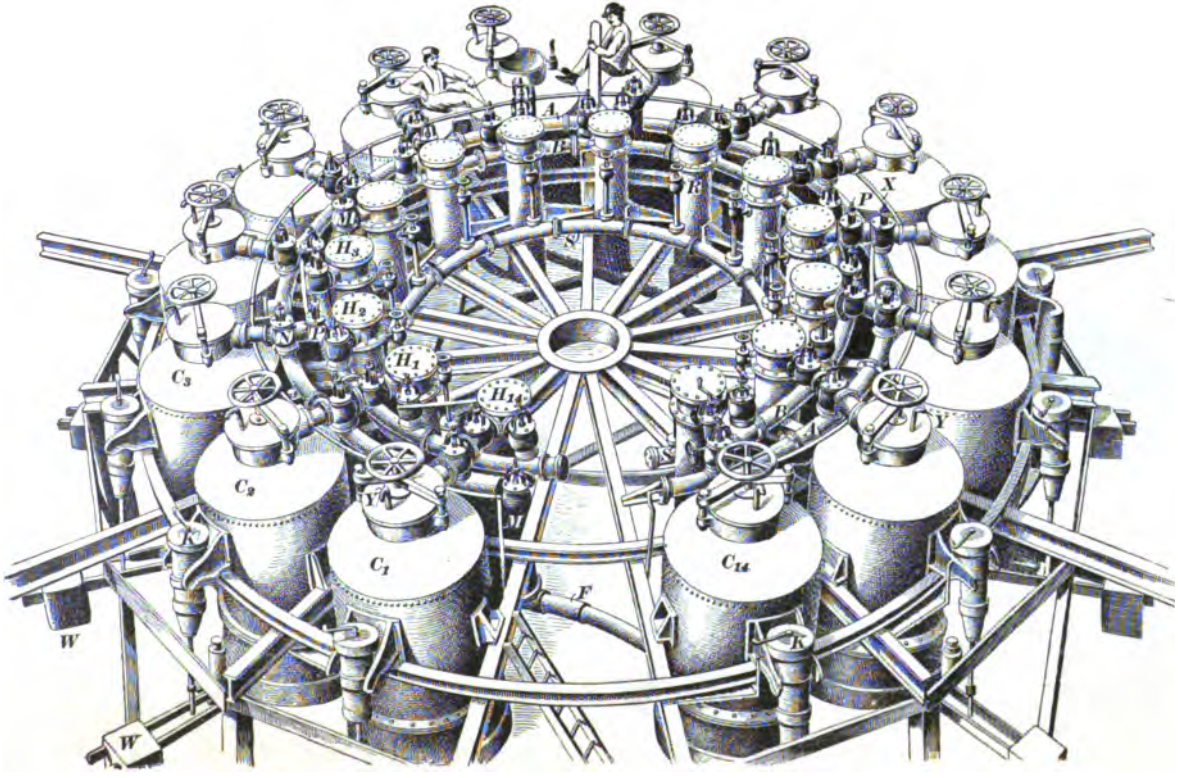


FIG. 7

so on. When the first diffuser is filled with water, or, as it may now be termed, *juice*, the direction of the current in this diffuser is reversed by a proper manipulation of the valves. The juice is passed up through the first heater into and downwards through the heater of the second diffuser; it enters the latter at the bottom and passes upwards as before.

There are two objects in admitting the juice, or water, at the bottom of the diffuser: (1) the juice passes through two heaters, thus exposing it to a large heating surface, and (2) on entering at the bottom of the cell, the juice drives the air and gases ahead of it and distributes itself uniformly throughout the cossettes. When the second diffuser is filled with juice, the direction of the current is changed in it, as in the previous vessel. When nine cells of a battery made up of twelve cells have been filled as described, a measured quantity of juice is drawn from the last, the circulation of the liquid being maintained by passing air or water into the first diffuser of the series. During these manipulations, the temperature of the juice in the various diffusers is regulated to meet certain conditions that will be described later.

After drawing a quantity of juice from the ninth cell into a measuring tank, the first diffuser is cut out of the series and opened. The pulp, or exhausted cossettes, that this diffuser contains is then discharged from it. If the battery is composed of more than twelve diffusers, all but three are filled before drawing a charge of juice or discharging the pulp from a diffuser. After the first cell is cut out, the second becomes the first of the series and is the next to be emptied. From this time on when a cell is filled with fresh cossettes and liquor, a charge of juice is drawn from it and the first diffuser is emptied. As just explained, the juice is always circulating through all but three of the diffusers; hence, the cossettes are in contact with the diffusion juice of various densities for about 1 hour and 20 minutes, or longer. The concentration of the juice increases from diffuser to diffuser; that of the first vessel contains only a small fraction of a per cent. of sucrose, and the juice of the last cell has a density of about four-fifths that of the normal juice.

24. The exhausted cossettes are removed from the factory by means of a screw conveyer or a drag, or they are flushed from it with water. The further treatment of this by-product will be considered later. Under favorable working conditions, this residue contains from .15 to .25 per cent. of sucrose, in terms of the weight of the beets.

There is a small loss of sucrose in the waste waters that come from the battery, and there is always some water or very thin juice left in the first diffuser after drawing the measured quantity of juice from the last vessel. This thin juice contains only a small trace of sucrose.

25. From the foregoing description of the operation of a diffusion battery, it will be seen that the theoretical conditions of the process are fairly well complied with in its practical application. Thus, the thin slices of the beets, which expose the cell membranes of the plant, serve to separate the beet juice from the water used in bathing the cossettes. The slices remain in contact with the liquid for more than an hour, or long enough for the principal crystalloid—sucrose—to pass through the membrane. As will be shown further on, the temperature of the battery is regulated so as to secure rapid dialysis without destroying the cell membrane. However, notwithstanding all the precautions, the juice obtained is quite impure, considering everything except sucrose as impurities, because many plant cells are ruptured by the slicer and many crystalloids besides sucrose are extracted.

As previously stated, a measured quantity of juice is drawn from the last diffuser. This juice is conducted to the next station of the factory for purification. The quantity of diffusion juice to be drawn depends on the quality of the beets and the amount of dilution of juice that manufacturing conditions will permit. Under favorable conditions, the density of the diffusion juice should be about four-fifths that of the normal juice. To obtain juice of this density, and at the same time exhaust the cossettes of sugar sufficiently, from 109 to 113 liters of juice should be drawn per 100 kilograms

of beets. This volume of juice is varied according to the richness of the beets, but is fairly constant during the greater part of the manufacturing season. This is due to a decrease in the volume of the normal juice of the beet as the latter increases in maturity. The juice is usually measured in a rectangular or circular tank, the batteryman recording the volume and temperature of the juice drawn on a blank form. The record of the volume is often kept automatically by the Horsin-Déon or some other recording device.

26. Temperature Conditions.—The control of the temperature at which the diffusion is conducted is of great importance. If the temperature is too high, the cell membranes of the beets break down, or are "cooked," and the pectic bodies pass into solution in the juice. As the dialysis is rapid at high temperatures and sluggish with cold solutions, the diffusion is conducted at as high a temperature as is practicable without affecting the stability of the cell membranes. Experience has demonstrated that the maximum temperature should not exceed 85° C. If the temperature is too high, the exhausted cossettes cannot be properly pressed for use as cattle food, and they may even pack in the diffusers and thus render their discharge from the battery very difficult. Perfectly sound beets withstand a higher battery temperature than those which have been stored a long time or have been frozen. M. François Dupont, President of the French Association of Sugar Chemists, recommends the following range of temperatures, in degrees centigrade, for the active diffusers:

Diffuser No.	1	2	(3, 4, 5, 6, 7, 8)	9	10
Temperature	40°	60°	76° to 85°	65° to 70°	40° to 50°

Diffuser No. 1 contains the exhausted pulp and No. 10, the fresh cossettes. The maximum temperature should be lower when treating beets of poor quality.

27. Remarks on the Conduct of the Diffusion.—In the event of a long stoppage of the manufacture, it is necessary to empty the diffusion battery. To accomplish this with little loss of sucrose, the usual procedure is as follows: Two charges of juice are drawn from the last diffuser, and the

pulp is then discharged from the first vessel; two more charges are drawn, and the pulp is then discharged from the second diffuser. This operation is continued, drawing one or two charges of juice from the last cell and each time discharging a diffuser. As the density of the juice decreases, the number of charges drawn per cell may be reduced.

Should the supply of beets be insufficient for operating the plant at its full capacity, the rate of slicing should be reduced and the battery should be run at a comparatively low temperature. For satisfactory work, the cossettes should be smoothly cut with sharp knives and should be thin but not too thin. The work should be conducted rapidly and with regularity, and the water supplied to the battery should be of good quality and free from bicarbonates, sulphate of calcium, or other substances that would deposit on the surfaces of the heaters, etc. It is common practice to conduct the work at the rate of 200 diffusers of cossettes per 24 hours.

PURIFICATION OF THE JUICE

28. Straining, or Depulping, the Juice.—As it is drawn from the diffusion battery, the juice contains large quantities of minute fragments of beet pulp. These must be removed prior to the carbonatation process, since the pectin bodies would be decomposed and, at least in part, be rendered soluble. The depulping, or straining, is accomplished either between the diffusion battery and the measuring tank or after the juice leaves the latter.

A common type of strainer consists simply of a metal basket with fine perforations, through which the juice is caused to flow. This basket is placed in a suitable receptacle and is protected from the air. It is removed from time to time for cleaning. In another type of depulper, finely perforated brass sheets are formed into pockets similar to the cloth bags of the mechanical juice fillers. The juice passes, under low pressure, from the outside of the pocket to the inside and leaves the pulp on the strainer, from which it falls when much of it has accumulated. The

pockets are placed in an iron box and are so arranged that the clean juice may be led off from the inside of them and the pulp removed as required.

29. Reheating the Juice.—As the juice leaves the diffusion battery, its temperature is from 40° to 50° C. This low temperature was formerly considered desirable at the beginning of the carbonatation, but the present practice is to heat the juice to about 90° C. The object of this heating, aside from the economy of fuel, is to coagulate a part of the albuminoid matters. The heating is accomplished by passing the juice through calorisators that are heated either by a part of the vapors from the first pan of the multiple effect or by those from the last pan on their way to the condenser. These vapors would otherwise be wasted.

The reheating of the juice is not generally practiced, and its utility from the point of view of the purification of the juice is questioned by competent authorities.

30. Purification of Juice by the Carbonatation Process.—The double-carbonatation process is always used in the purification of the beet juice. This process is divided into the *carbonatation* and the *saturation*, or *second carbonatation*. In the carbonatation, a large quantity of lime, usually from 2½ to 3 per cent. of the weight of the beets, is added to the juice, and then all but a small quantity of it is precipitated with carbonic acid. After the removal of the precipitate, a small quantity of lime—about .5 per cent. of the weight of the beets—is added to the juice, and is then precipitated with carbonic acid, together with that left in the previous operation. In this second operation, all the lime is precipitated; hence the use of the word *saturation*. After a brief discussion of the objects of the liming and carbonating, the carbonatation process itself will be described more in detail.

31. Purposes of the Liming and Carbonatation. The lime added to the juice combines with the free acids of the latter and also with those in weak combination; it also forms a soluble saccharate with a part of the sucrose and it precipitates the silica, the pectic bodies, and the albuminoids

in part. The lime compounds, with the acids of the juice, consist of both soluble and insoluble salts. The combination of the alkalies, sodium, potassium, and ammonium are broken up and their carbonates are formed. Lime salts are formed in part also from the decomposition products of the glutamine and asparagine. During carbonatation, the soluble alkalies are converted into carbonates, as is also the lime. The calcium carbonate entrains and also occludes many of the mechanical impurities and carries them down with itself.

The precipitation of all the lime in carbonatation is avoided, so as to prevent insoluble combinations of the lime and impurities from being broken up. After these compounds have been removed by filtration, in the second part of the process, or the saturation, all the lime is removed by precipitation.

The carbonatation process employed for beets corresponds to the defecation process used for sugar cane, and its purpose is to remove, so far as is possible, all the non-sucrose from the juice, preparatory to the crystallization of the sugar.

32. Carbonatation Process.—After reheating the juice to 65° or 75° C., a quantity of quicklime, in lumps, equal to from 2 to 3 per cent. of the weight of the beets from which the juice was derived, is slaked in the juice; or, an equivalent quantity of milk of lime may be used, in which case the temperature of the juice may reach 85° C. The lump quicklime is generally used in Germany, while slaked lime is used in other European countries and the United States. If the factory is equipped for the recovery of the sugar from the molasses by a lime saccharate process, the saccharate is used in liming the juice.

After liming the juice, carbonic-acid gas is forced into it until the alkalinity is reduced to the equivalent of 1 to 1.6 grams of calcium oxide per liter of juice, as indicated by phenol phthalein. The attendant judges this point by examining a test sample held in a spoon. The precipitate at this time appears granular and the liquid yellow. This

test is frequently verified by rapid chemical analysis, in which a standardized sulphuric acid, with phenol phthalein as an indicator, is used. Other indicators, for example, coralline, may be employed, but the apparent alkalinity varies somewhat with different indicators. The experienced workman learns to note the approaching close of the operation by the appearance of the precipitate and the sound of the escaping bubbles of gas. As, under favorable conditions, the carbonatation lasts only about 10 or 15 minutes, the tests must be made very rapidly. Excessive carbonatation, resulting in impure juices, must be avoided. During the carbonatation, large quantities of foam are formed. This may be beaten down by jets of steam or by the application of tallow or oil in small quantities. Very deep tanks are often employed so as to avoid the use of steam or oil. These tanks are frequently more than 20 feet in depth.

When the test indicates that the juice has been sufficiently carbonated, the juice is quickly heated to 90° C. and is then filtered through presses.

33. Saturation.—**The second carbonatation, or saturation,** is conducted as follows: To the filtered juice from the carbonatation, from 2 to 4 pounds of lime per 100 gallons is added. Carbonic-acid gas is again forced into the juice until all the lime present that is precipitable by this reagent has been saturated. The juice should remain alkaline after this second carbonatation, owing to the presence of other alkalies than lime. The end of the reaction may be ascertained by a titration with a standardized sulphuric-acid solution, or less accurately by means of phenol-phthalein test papers. The alkalinity that should remain varies with the beets, but is usually equivalent to .02 gram of lime per liter of juice. When the saturation is completed, the juice is heated to boiling and is then filtered through filter presses. If this carbonatation is not carried far enough, still leaving an excess of lime in the juice, difficulty may be experienced in boiling the liquors in the vacuum pan. The evaporation will progress slowly or not at all.

The process has been described as it is usually applied. It is sometimes modified by using other quantities of lime or other temperatures, or by carrying the carbonatation somewhat further. The filter-press work is usually a guide in conducting the carbonatation. If the press cake is very white and chalky, more lime than necessary has been used. With insufficient lime, the filtration is sluggish and the press cake is pasty. The proper quantity of lime yields a grayish-colored cake, which is firm and readily washed.

34. Production of Lime and Carbonic Acid.—The carbonic acid for the precipitation of the lime and the lime itself are obtained by calcining limestone in a special type of lime kiln. The kilns in former times were conical and were constructed entirely of brick with a lining of firebrick. In the present construction, a large iron casing, the frustum of a cone, is supported on columns and at its lower part is suspended a bottom section that is also the frustum of a cone. These sections are fastened together at the bases, forming an enlarged section of the kiln. The upper section terminates at the top in a feed-opening for coke and limestone and in an emergency chimney. The lower end of the bottom section is open and near the ground, on which is built a conical distributor. The iron casing is lined with firebrick.

In starting the kiln, limestone is piled on the distributor until it closes the lower opening; then kindling is thrown into the kiln at the feed-door, and this is followed by alternate charges of coke and limestone in the proportion of 1 volume of coke to 4 or 5 volumes of stone. The charges should be small, so that the fuel and stone may be well mixed and distributed. The fire is started and is maintained by natural draft through the chimney until the stone begins to decompose and the gas is required in the factory. A large pipe leads from near the top of the kiln to the gas washer and thence to the carbonatation tanks.

The function of the washer is to cool the carbonic-acid gas and to remove impurities. In one of the usual types, the gas flows in a direction opposite to a stream of water that falls in

cascades from shelf to shelf of the washer. About 24 hours after lighting the kiln, carbonic-acid gas may usually be drawn from it through the pipe and washer by means of the gas pump and be forced into the juice in the carbonatation tanks. The kiln should supply a gas containing about 30 per cent. of carbonic acid and should be practically free from carbonic oxide.

At intervals, lime is drawn from the kiln, which is frequently charged with stone and coke in the proportions already stated. The type of kiln described is used by several large American factories and is one of the most efficient.

35. Carbonatation Tanks.—Figs. 8 and 9 are illustrations of an E. H. Dyer and Company **carbonatation tank**, in which *a* represents the inlet valve for the diffusion juice; *b*, the outlet valve for the finished, or carbonated, juice; *c*, the valve for the entrance of the carbon dioxide, which is forced into the juice through the perforated pipes *d*; *e*, *e'*, the steam valves supplying steam for the heating coils *f*, and for the perforated pipe *g*, the latter being used for reducing the foam on the juice; *i*, the sampler for drawing test samples of the juice, which may be obtained without opening the tanks by simply pressing down on the handle *j* and holding a dipper under the spout *k*; *l*, a thermometer; *m*, a sliding cover; *n*, a manhole in the bottom of the tank for cleaning purposes; and *o*, an opening to the vent pipe, which liberates superfluous gases to the outside air through a stack.

The gas-distributing pipes are often so arranged that they may be readily removed from the tank for cleaning. The lime scale that forms on these pipes and obstructs the perforations can be easily loosened by heating the pipe in a blacksmith's forge, and the scale that accumulates on the walls of the tank and the heating surfaces can be removed either by scraping or by boiling acidulated water in the vessel.

Before workmen enter a tank, it is always necessary to ascertain whether the tank is free from carbon dioxide and monoxide.

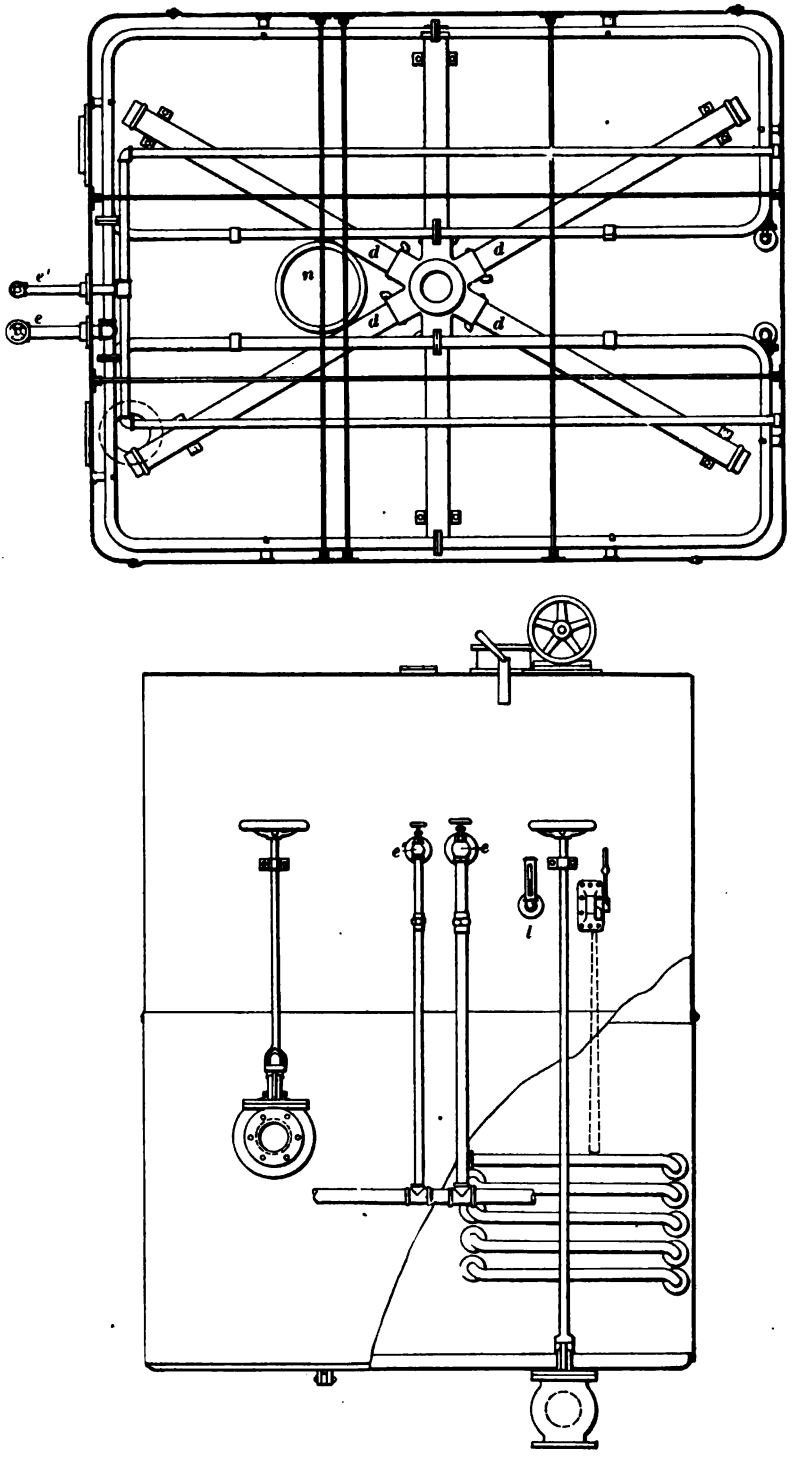


FIG. 8

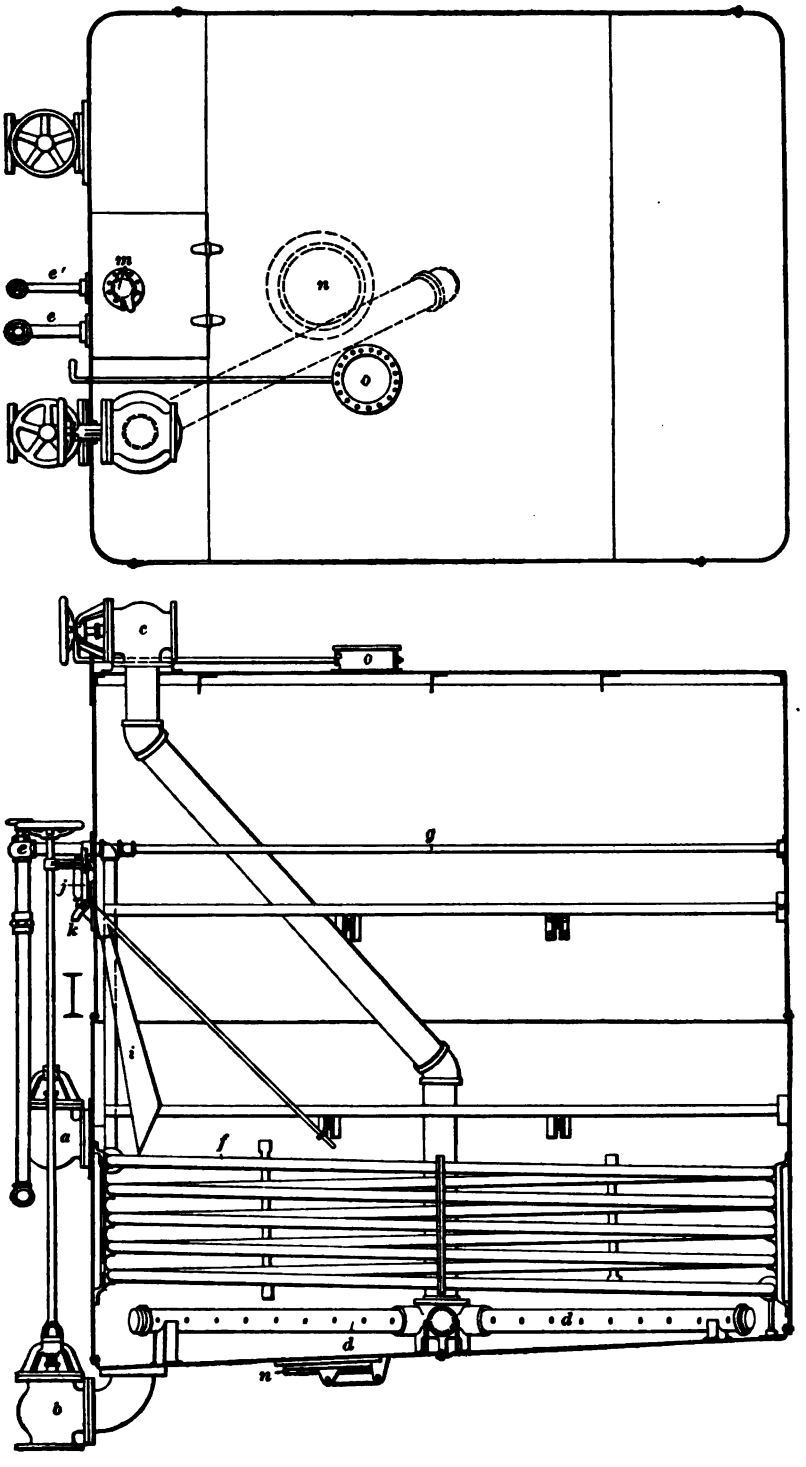


FIG. 9

The carbonatation and saturation may also be conducted in a continuous carbonator; that is, an apparatus into which limed juice is constantly flowing and from which there is a continuous stream of carbonated juice. Such apparatus, however, is seldom used.

36. Sulphuring.—Sulphurous-acid gas is applied in beet-sugar manufacture, as in the cane-sugar industry, to obtain a white product or to increase the yield of sugar, and the process is termed **sulphuring.**

Practice differs somewhat as to the stage of the manufacture at which to use the sulphurous acid, but usually it is the filtered juices from the saturation, or second carbonatation, that are sulphured.

The juice may be sulphured either with or without again liming it, but usually no lime is added. In the latter case, all the lime is not precipitated in the second carbonatation; the carbonating is stopped when the alkalinity is equivalent to about .5 gram of calcium oxide per liter, and the juice is then filter-pressed. From the presses, the juice flows into sulphuring tanks, where sulphurous-acid gas is passed into it until the alkalinity is reduced to the equivalent of about .1 gram of lime per liter. The quantity of lime left from the second carbonatation or that added before sulphuring must be sufficient to insure the proper filtration of the juice. After sulphuring, the juice is heated to the boiling point and again filtered. Special filter presses or mechanical filters are used for the filtration.

Sirups are sulphured in some factories, as is also the massecuite, while boiling in the vacuum pan. The sulphuring of massecuites, however, is unusual.

37. Sulphur Stoves.—There are two general types of **sulphur stoves,** namely, *closed stoves*, into which air is forced by a pump; and *open stoves*, in which the draft is induced either by an ejector in the chimney or by a pump. The closed stoves deliver the gas under pressure and are used almost exclusively in beet-sugar manufacture, while the second type is used in the cane-sugar industry.

38. The **closed stoves** are of various forms, but in cross-section they are usually the shape of an inverted **U**. This type of stove is made of thick cast iron, as is also the gas pipe leading from it. At the front of the stove is a door that forms a tight joint when closed and serves for the introduction of sulphur or for cleaning the apparatus. The sulphur is burned in a cast-iron pan. Both the stove and the gas pipe are water-jacketed for the cooling of the gas. This is advisable, because sulphurous acid is decomposed into its elements at high temperatures. Air is forced into the stove by a compressor and is usually delivered at the front of the sulphur pan or under it. The stoves are often provided with baffle plates so as to prevent sublimed sulphur from reaching the gas pipes; they are also often arranged for charging with sulphur while in use.

To operate a closed stove, the pan is filled with sulphur and a piece of red-hot iron or a lighted fragment of burlap bagging saturated with sulphur is dropped into the pan; the door is then closed tightly and the air compressor is started. The quantity of gas produced is regulated by altering the speed of the air compressor.

39. The **open stoves**, which employ an induced draft, are described in *Manufacture of Sugar*, Part 1. Such stoves, as already mentioned, are used almost exclusively in cane-sugar manufacture.

40. Sulphuring Tanks.—The **sulphuring tanks** do not differ materially from those used in the carbonatation. As there is no frothing of the juice during sulphuring, the tanks may be almost entirely filled with juice. The valves for admitting the gas are made of hard rubber or of a lead composition; if made of some other material the acid would be liable to destroy them quickly.

Instead of using tanks similar to carbonatation tanks, a continuous process of sulphuring is often employed. In one form of continuous apparatus, a tank is divided into five compartments by partitions so arranged that the juice passes under the first of these into the second compartment and

overflows into the third, and so on. Sulphurous-acid gas is forced into the juice in each compartment through a perforated pipe. There is sufficient travel for the juice to insure thorough sulphuring. The quantity of gas used is regulated by means of the air compressor. The tanks may be of wood and are preferably located at the top of the factory, to permit the escape of the gas, provided none is absorbed. After sulphuring the juice, it is heated to boiling, preliminary to its filtration.

FILTRATION OF THE JUICE

41. Filter Presses.—The juices from the first and second carbonatations and often the sulphured juices are filtered through cloth in **filter presses.** Formerly, the juice from the first carbonatation was separated from the precipitates by decantation. Filter presses are of two general types, namely, *center-feed presses* and *frame presses.*

42. The **center-feed press** is now comparatively little used. It usually consists of a series of recessed rectangular plates of cast iron, with a large central opening in each and also a drain cock at one of the lower corners. Both sides of the plate have either grooves or grooves covered with a perforated metal plate. Filter cloths having an opening corresponding to the central ones in the plates are placed over the latter and are clamped to them at the opening. The plates, supported on a suitable frame, are clamped together by means of a powerful screw, and the cloths themselves form tight joints between the plates. The central openings form a channel that connects with the recessed space formed by the plates, and through this the juice and the precipitates are forced into the press. The lime precipitate accumulates in the space between the plates, and the juice passes through the cloths into the grooves leading to the drain cocks and juice troughs. The precipitates form a hard press cake, which is removed from time to time by opening the press.

43. In Fig. 10 are shown many details of a **frame press,** and in Fig. 11 an installation of this type of press is illustrated. The press consists of a series of grooved plates and frames that alternate with one another. At *a*, Fig. 10, is a frame and at *b* a plate. The vertical grooves of the plate at their lower ends connect with a horizontal channel and through an opening with a juice cock *j*. The plates and frames have side lugs that rest on beams *c*. At *d* and *e* are shown the head-piece and the tail-piece, respectively. The screw for clamping the plates and frames together is shown at *f*. A sheet-iron pan, or tray, *k* serves to catch the juice that drips from the cloths or leaks from the press. The juice trough *g* has two outlets *h* and *i*, one for filtered juice and the other for wash water. At the lower part of each plate and frame, as shown in Fig 11, there are two lugs, one on each side. In each of these lugs is an opening, forming, in one side of the press, the juice and precipitate channel, and in the other, the wash-water conduit. The juice channel connects directly with each frame, and the water conduit with the grooves of every alternate plate, that is, with the "water plates." The cloths in this type of press have no holes in them. The joints between the channel lugs shown in Fig. 11 are made with cloth "stockings," which have openings in them that correspond with those in the lugs. In the Kroog press of this type, these joints are made with rubber rings.

In "dressing" the press, the cloths are laid over the plates so as to form joints between the plates and the frames, as shown in Fig. 11, and the clamp screw *f*, Fig 10, is then tightened.

44. Referring to Fig. 11, when the press is in operation, the juice is admitted to it under pump, or monte-jus, pressure through the valve *l* and is distributed through the channel into the frames. The juice filters through the cloth, leaving the precipitate in the frame in the form of a compact cake. When the press is filled with precipitate, the juice cocks on the water plates are closed, as is also the valve *l*, and water is admitted to the press through the valve *m*. The water

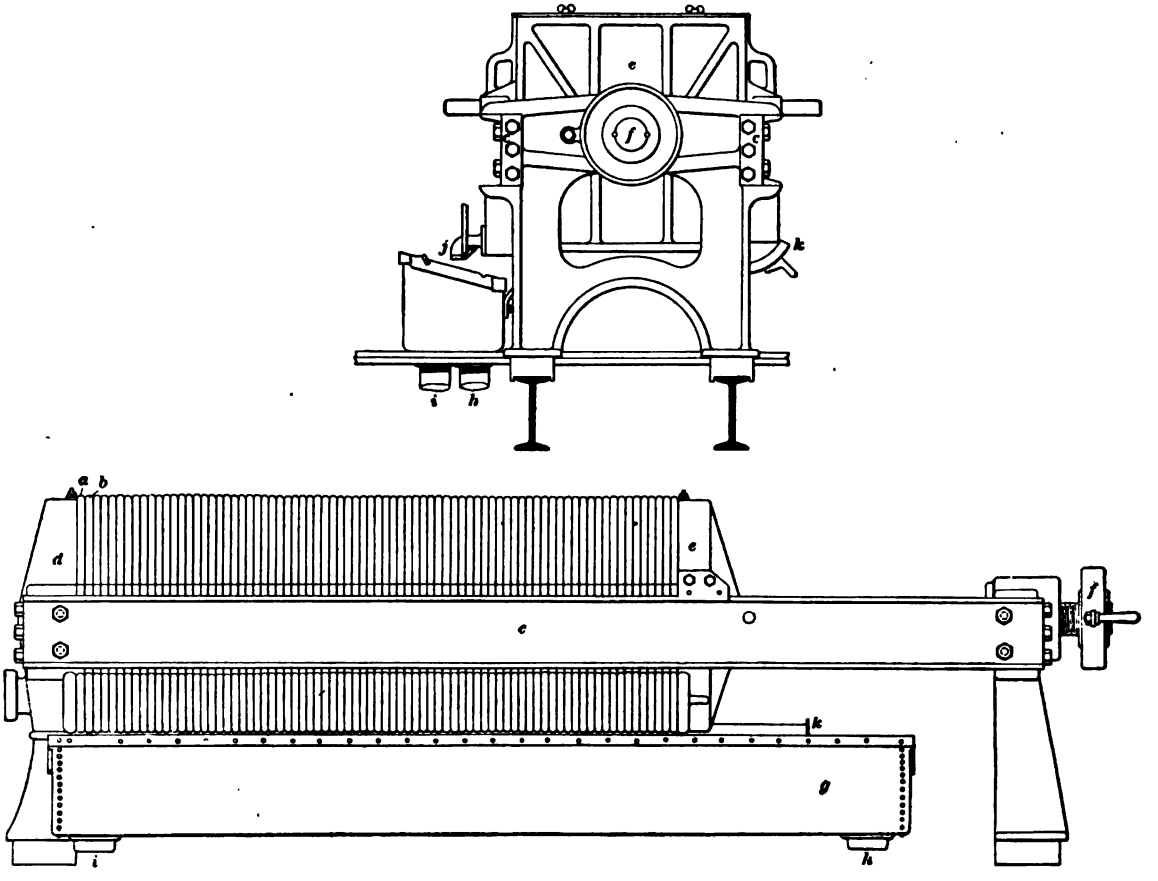


FIG. 10

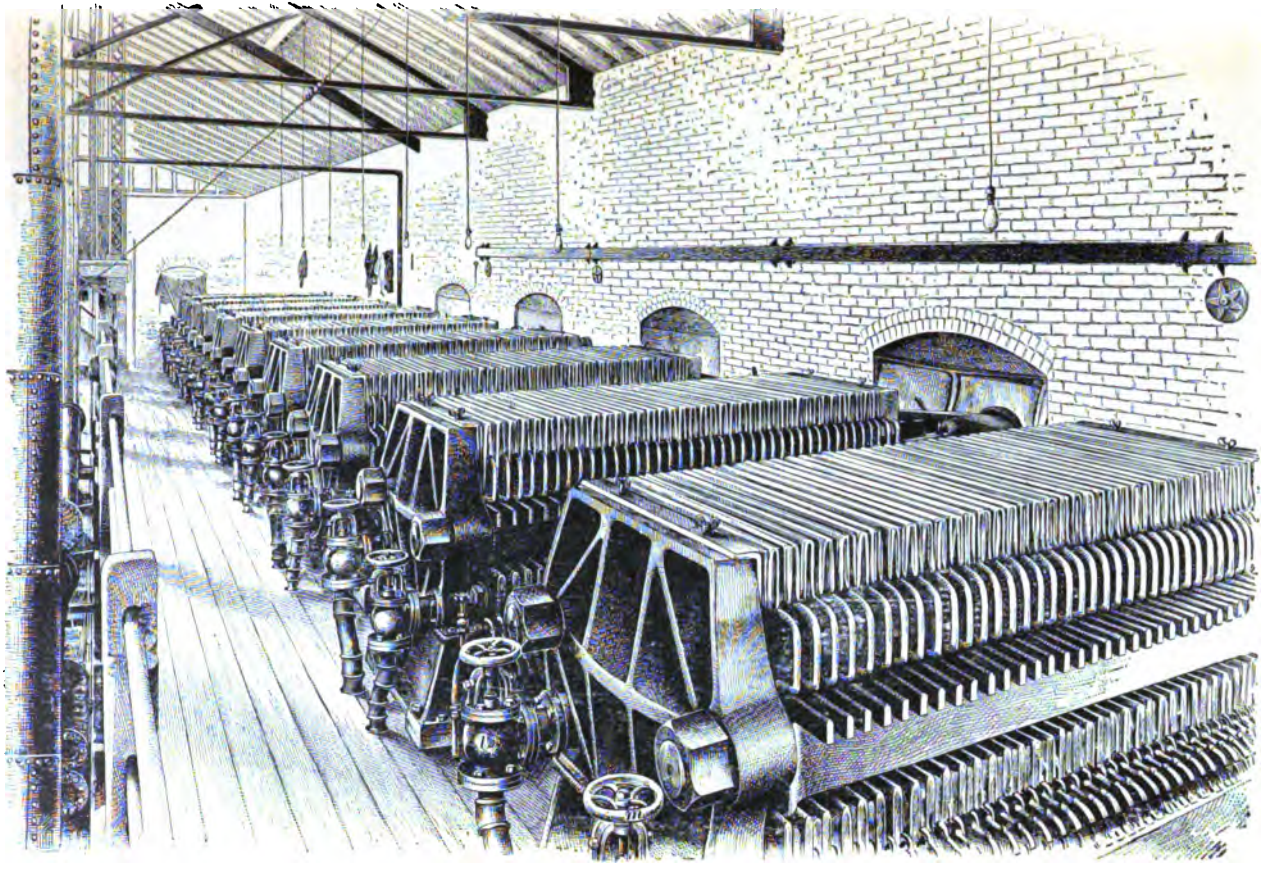


FIG. 11

fills the spaces behind the cloths, passes into the press cake, and drives the juice ahead of it and through the open juice cocks to the trough. The frame presses often are designed to produce a cake about 1¼ inches in thickness.

45. In the Gallois method of washing the cake in a center-feed press, a three-way cock is used. The press is first nearly filled with the precipitates; then the cock is moved a little farther, water and juice are admitted, and finally the cock is turned to admit water only.

In washing the cake in the frame presses, there is comparatively little dilution of the juice, but the contrary is the case with the Gallois device. With both processes, either cold or hot water may be used and apparently with equal efficiency. The press cakes were formerly steamed, instead of being washed with water, but this method has been abandoned on account of the damage to the cloths. The washing of the press cake reduces its sucrose content to about 1 or 2 per cent. In many factories, instead of washing the press cake, it is reduced to a thin cream with warm water and again filter-pressed.

46. After washing the cake, the presses are opened and their contents are discharged either into a car, for removal from the factory, or into a mixer, where they are reduced to a cream with water and then run into a settling pond. At the close of the season, the water is decanted from the precipitate, which soon dries and may be removed with scrapers. Certain American factories that use the Steffen lime-saccharate process calcine the press cake in Bauer's rotary kiln and recover the lime and carbonic acid for further use.

Jute or cotton cloths are used in dressing the presses. These cloths are usually changed about once a week, and the soiled cloths, after being washed, may be used again.

Special presses having very shallow recesses are often used for filtering sulphured juices, although mechanical filters with cloth pockets are usually preferred.

47. Mechanical Filters.—There are many types of the so-called **mechanical filters**, but they may be divided

into three general classes, as follows: (1) Filters in which the liquor passes from the inside to the outside of a bag, or pocket; (2) filters in which the precipitates collect on the outside of a bag and the clear juice is drawn off from inside of it; and (3) filters in which sand or other granular material is used as a filtering medium.

48. Filters of the first class are seldom used in sugar factories. Such filters are employed in refineries, and will be described in connection with the methods of refining.

49. Filters of the second class are very extensively employed in the filtration of both juices and sirups. The *Danek* type of filter of this class is used by many factories in America. This filter consists of an iron box, in which are suspended a number of cotton bags, or pockets. Each bag is slipped over a metal sheet, in which are punched rectangular openings. The metal is not entirely removed from the openings, but is formed into a bow-shaped piece, which keeps the cloth away from the plate. The openings are very close to one another, and the bow-shaped guards keep the pocket distended and permit a free circulation of the liquid within the bag. The upper end of the metal sheet is attached to a longitudinally slotted pipe, one end of which is closed while the other projects beyond the bag for the delivery of the juice. The pocket is clamped to the pipe in such a way that nothing can enter it except through the pores of the cloth. On one side of the filter box are openings, into which the delivery pipes of the pockets enter and connect with discharge cocks on the outside. These cocks deliver the filtrate into a gutter. The delivery pipes have inclined planes at their ends, opposite the cocks, and these planes bear on corresponding planes in the side of the filter box, so that the weight of the filtering element itself is sufficient to press the pipe against suitable packing and thus form a tight joint. The part of the chamber below the pockets is deep and serves to collect the accumulations of mud. The filter box has a counter-poised hinged cover that closes it tightly. A pipe enters near the bottom of the pockets for the admis-

sion of cloudy juice, and a larger pipe leaves the box at the bottom for the discharge of accumulated mud. When the box is open, the filtering elements may be readily lifted from it for cleaning purposes; and they may be as easily replaced owing to the inclined planes, which force them into position. A suitable device prevents the elements from coming in contact with one another or from being lifted by the current of liquor.

50. In using the filter, the liquor is admitted to the box from a tank under a head of 2 to 3 meters. The liquid passes into the pocket, on the outside of which the mud accumulates and then falls to the bottom of the box. The juice passes into the slotted pipe and is drawn off at the cocks. The mud is removed from time to time through the pipe at the bottom of the box and is filter-pressed. Low pressure is essential in the filtration with this type of filter, to prevent the mud from compacting on the cloth and rendering it impervious to the liquors.

Several filters of this type are used that differ from the Danek only in the method of attaching and distending the pockets or in other mechanical details. The pockets are usually 70 cm. × 70 cm. in size, and vary in number from twenty upwards.

51. Filters of the third class have found a wide application in the sugar industry. Sand is the usual filtering medium. The sand filters most generally employed are the *Breitfeld-Danek* and the *Abraham*.

52. The **Breitfeld-Danek filter** consists of a large, rectangular, iron box containing a shallow layer of sand on a suitable support. A pipe with a regulating valve maintains the level of the liquor at about 25 centimeters above the sand, and the filtered liquid is drawn off below the sand. Pipes and valves are provided for washing the filtering medium. When the filter is in use, the liquid percolates through the sand and its suspended impurities penetrate the filtering medium to a depth of only a few centimeters. These filters may be used for the filtration of juices, sirups,

and molasses. The liquor to be filtered must be heated to near its boiling point.

When the filter becomes clogged with impurities, the sand is first washed from above with water, to recover the sugar it contains, and then a strong stream of water is forced into it. The sand is lifted by the water and the attrition of the grains removes the precipitate from them. The muddy water overflows into a pipe leading to the sewer. When the sand has been washed clean, the water is shut off and the sand settles back into place, ready for use.

53. The **Abraham filter,** as recently constructed, consists of a vertical iron cylinder, inside of which is a much smaller perforated metal cylinder. The outer cylinder is filled with a large number of superimposed flaring rings, with the large diameters of the rings turned upwards. Pipes with regulating valves communicate with the outer cylinder, to convey liquor into the filter, and with the inner cylinder, for the exit of the filtrate. The filter has a removable cover, and at the bottom, which is conical, there is a manhole with its cover. With the exception of the annular space surrounding the rings and the interior of the inner cylinder, the filter is filled with sand. When this filter is in use, the liquor to be filtered enters the outer cylinder, and, owing to the presence of the flaring rings, is distributed to the outside of the column of sand. The liquor passes through the filtering medium into the perforated cylinder and thence to the filtered-liquor tanks.

The sand is removed from the filter for washing. Other similar filters are arranged for washing the sand in the same manner as in the Breitfeld-Danek. The sand filters of Hyatt and Jewell, which use water, present familiar examples of this method of washing sand. The sand for use in filters should preferably be very sharp, with grains of uniform size. Other materials, such as cork, sawdust, asbestos, lignite, etc., are used instead of sand in certain mechanical filters.

54. Remarks on Filtration.—As already stated, filter presses are used for filtering the juices from the carbonatation, since these juices contain large quantities of gran-

ular precipitates. The sulphured juices, and sirups, on the contrary, are filtered through mechanical filters, because these liquids contain fine precipitates that would quickly obstruct the pores of the cloths under the pressure used with presses. The ease with which the filtering medium can be washed, and its small cost and indestructibility, render sand filters very advantageous. There is a small loss of sand, however, in the wash waters. Filters are sometimes used for the filtration of the liquors while in transit from vessel to vessel of the multiple-effect evaporator.

Bone black was formerly employed in all beet-sugar factories for the filtration of the juice and sirup. This material, however, is no longer used, sulphurous acid for bleaching and mechanical filtration having displaced it.

CONCENTRATION OF THE JUICE TO A SIRUP

55. Multiple-Effect Evaporators.—The concentration of the juice is accomplished in **multiple-effect evaporators** of various types. The standard type of evaporator previously described is also used in beet-sugar manufacture, but perhaps not so generally as the *Welner-Jelinek* and evaporators of similar design.

56. A modified **Welner-Jelinek quadruple effect,** manufactured by E. H. Dyer and Company, is shown in Figs. 12 and 13. The principle of the multiple effect and the method of operating it have been previously described. Therefore, only a general description of a modified Welner-Jelinek apparatus will be given. The effects, or pans, are rectangular cast-iron vessels. The heating surface is composed of horizontal brass or copper tubes of small diameter that enter the heads, or distributing steam chambers, *a* and *b*. In this type of multiple effect, the steam circulates through the tubes, whereas in the standard evaporator, the juice fills the tubes and the steam surrounds them. The scale produced by the precipitation of certain impurities in the juice forms on the outside of the tubes. This scale

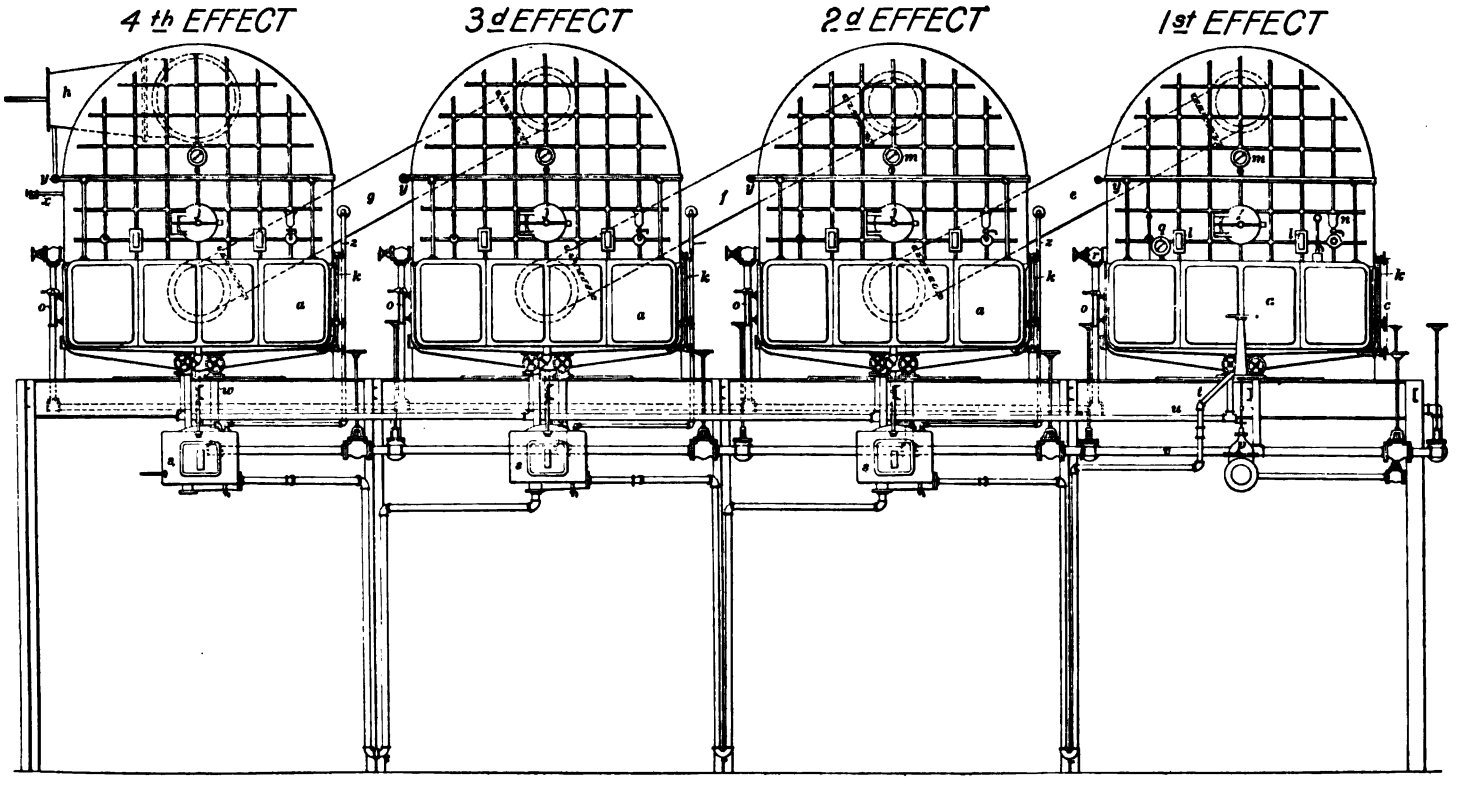


FIG. 12

can be removed either by spraying cold water on the tubes after heating them or by boiling caustic soda in the pan and following this with dilute hydrochloric acid. Scraping is also resorted to in cleaning the tubes.

In the evaporation of beet juices, there is a considerable evolution of ammonia arising from the decomposition of the nitrogenous constituents. The pipes *y* leading from the

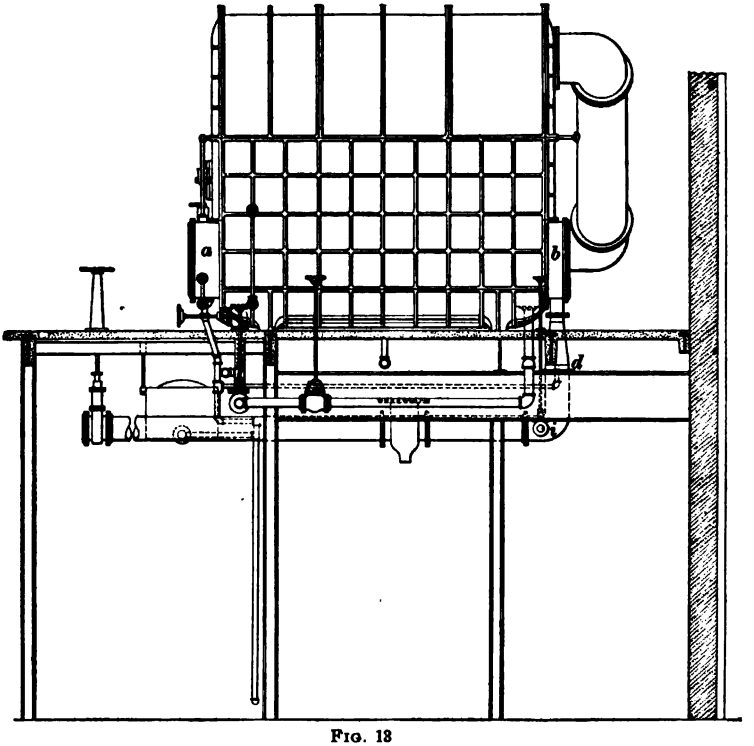


FIG. 13

steam chambers to the vapor pipes are for the purpose of removing this ammonia. The ammonia rapidly destroys the copper tubes, especially those in the upper rows. In the standard type of evaporator, the ammonia attacks the upper ends of the tubes.

57. In the following list, the parts of the apparatus indicated by letters in Figs. 12 and 13 are given:

a, *b*, chambers for the distribution of the steam and the collection of the water of condensation.

c, line indicating the depth of the juice space.

d, main for bringing exhaust steam from the engines and pumps to the heating surface of the first effect.

e, *f*, *g*, vapor pipes of the first, second, and third effects.

h, vapor pipe of the fourth effect. This pipe leads to the condenser.

i, live-steam pipe, for use when cleaning the heating surfaces or when starting the apparatus for heating the liquors.

j, manholes, to give access to the effects for cleaning or for repairs.

k, gauge glasses for juice. The small gauge glass below *a*, Fig. 13, is for noting the quantity of the condensation water remaining in the vessel.

l, eyeglasses, for watching the evaporation.

m, vacuum gauges.

n, butter cup, for injecting oil or grease so as to reduce frothing.

o, cups for drawing and examining samples of the liquors. The test samples are usually drawn only from the last effect.

p, main steam valve.

q, steam gauge.

r, water valve.

s, steam traps.

t, pipe-connecting pans and traps.

u, wash-out pipe.

v, pipe to convey liquor from pan to pan. This pipe is used in connection with charge pipes, not shown in the figures.

w, pipe leading from the fourth effect to the sirup pump, for the removal of finished liquor. This pipe is also connected with all the effects, for use in emptying them.

x, pipe connected with the sirup pump, for equalizing the vacuum in the last effect and the pump.

y, pipes for the removal of the ammoniacal vapors from the steam chambers.

z, pipes connecting the traps and vapor pipes.

58. Purification of the Sirup.—As it comes from the multiple effect, the sirup contains many impurities in suspension that must be removed. This is accomplished by settling and decantation or, preferably, by filtration. Mechanical filters that use either cloth or sand are employed for this filtration.

Prior to or after the filtration, the sirup is sometimes sulphured for bleaching purposes; it is again filtered after the sulphuring. A very small quantity of lime is added to the sirup before sulphuring, but this is not the usual practice. The experience of sugar makers has demonstrated that if sirups have been sulphured or have been produced from sulphured juices, they will boil freely in the vacuum pan, with a ready crystallization of the sugar.

MANUFACTURE OF SUGAR

(PART 4)

MANUFACTURE OF BEET SUGAR
(Continued)

CRYSTALLIZATION OF THE SUGAR

1. The methods of boiling sugar for use in the cane-sugar industry, which were previously described, are also employed in beet-sugar manufacture. The range of the apparent coefficients of purity, however, is higher in beet work. The high coefficient of purity of the molasses in beet work is due to the fact that the large quantity of mineral matter contained in this product prevents the crystallization of the sugar.

2. Crystallization in the Vacuum Pan.—As just stated, the methods of boiling beet sugar are identical with those used in cane-sugar factories. The sirup is first concentrated in the vacuum pan by evaporation until it is saturated with sugar; then, either by lowering the temperature of the liquor or by continuing the evaporation, supersaturation is produced and crystals are formed. The boiling is continued and sirup is added from time to time as the water evaporates, care always being taken to avoid supersaturation. The sugar in the sirup deposits itself on the crystals already formed, and by the time the pan is filled, these crystals will have grown to a suitable size for commercial sugar. The *masse-cuite*, as the mixture of sugar and molasses is now termed, is

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evaporated to a water content of 5 or 6 per cent. and is then discharged either into the mixer for the centrifugals or into crystallizers. This process is described in detail in *Manufacture of Sugar*, Part 1, to which reference should be made.

In case the saturation, or second carbonatation, of the juice, has not been properly conducted, thus leaving an excess of lime in the carbonated juice, the boiling of the strike will progress very slowly and perhaps not at all. In this event, the excess of lime must be saturated with a dilute acid—muriatic, for example—and the carbonatation must be carefully supervised.

3. A vacuum pan designed for use in beet-sugar manufacture is shown in Fig. 1. Following is a list of the apparatus indicated by the letters in the illustration:

a, body of the pan.

b, condenser. In this part of the apparatus, the vapors from the boiling liquor meet a current of water and are condensed, thus producing a vacuum. The air that enters the pan by leakage or with the sirup is removed by the vacuum pump, which is connected with the condenser by means of a pipe, not shown in the illustration.

c, the leg pipe for carrying off the condensed and condensing water.

d, tank for holding water to seal the foot of the leg pipe. The water is conveyed to the sluices by means of an overflow pipe *d'* and is used for floating beets into the factory to the slicers.

e, thermometer provided with a long stem, which projects into the pan to below the level of the proof stick *u*.

f, vacuum gauge.

g, butter cup, for injecting oil or tallow into the pan when required to reduce foaming of the boiling liquor.

h, eyeglasses, for inspecting the contents of the pan.

i, water or steam pipes, for cleaning the inside of the eyeglasses.

j, charge valve, for drawing sirup or molasses into the pan. This valve connects with a pipe that is located inside of the

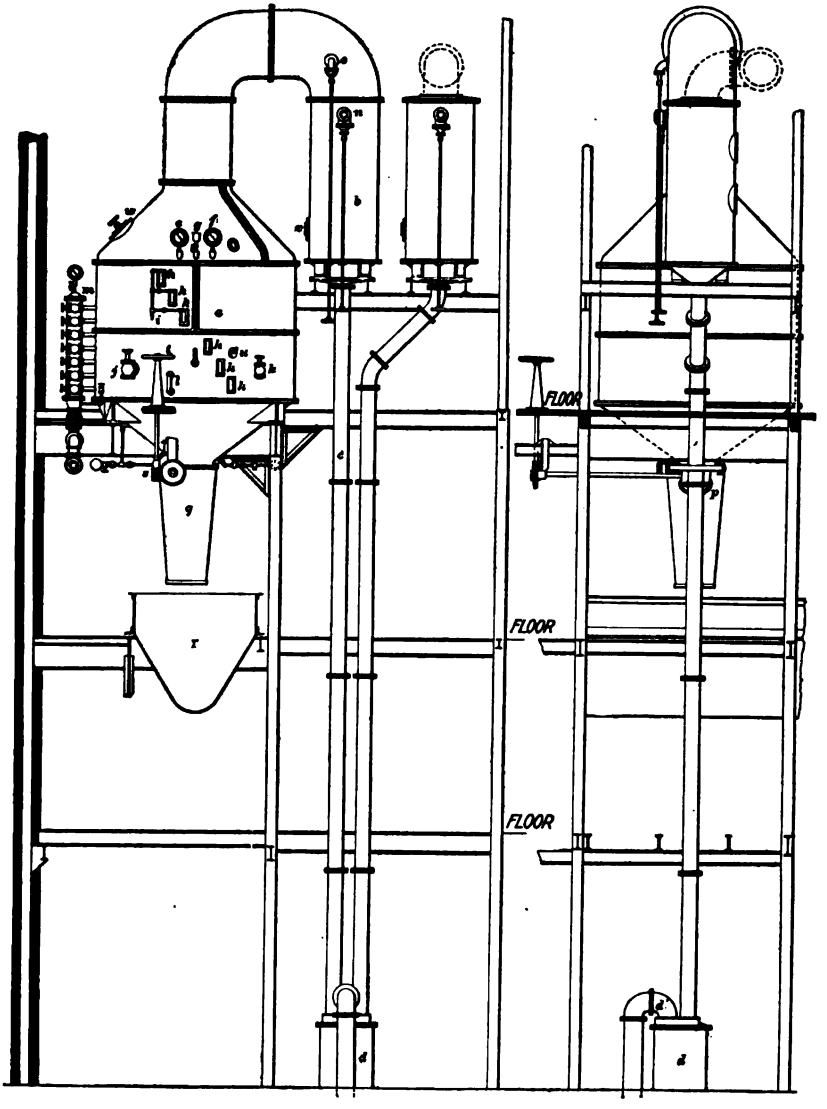


FIG. 1

pan. This pipe is often carried down outside the pan and should deliver the sirup near the foot-valve (*p*).

k, valve to admit water to the pan for cleaning, etc.

m, manifold, for distributing steam to the copper coils that form the heating surface of the pan.

n, condensing- or circulating-water valve.

o, break-vacuum valve.

p, foot-valve, or strike valve, for discharging the massecuite.

q, spout to direct the massecuite into the mixer or canals leading to the crystallizers.

r, mixer, for preparing massecuite to be purged in the centrifugals.

s, worm-gear, for manipulating the foot-valve.

t, wheel, for operating the gear *s* in opening and closing the foot-valve.

u, proof stick, for removing test samples of massecuite.

v and *w*, manhole covers. The removal of these covers gives access to the pan and condenser for cleaning and repairing.

At the right of the condenser *b* and the leg pipe *c* are shown another condenser and another leg pipe. These belong to another pan or to a multiple effect, which also utilizes the hotwell *d*.

The pan shown in Fig. 1 has no save-all to catch the sirup entrained with the vapors. This device should never be omitted. A pan having a save-all *B* is shown in Fig. 3, *Manufacture of Sugar*, Part 2; also in Fig. 8, *Manufacture of Sugar*, Part 2, a very efficient form of save-all is shown at *B* and *C*. This form of save-all is often used in English pans.

4. Crystallization in Motion.—The process known as **crystallization in motion** originated with Wulf, in Germany, in 1884, and the first practical apparatus for use was devised by Stammer and Bock. Crystallization in motion is now used in all beet-sugar factories. The process consists essentially in forming the crystals of sugar either in a vacuum pan or in a crystallizer and keeping them in motion until the crystallization is completed. It is always preferable to carry

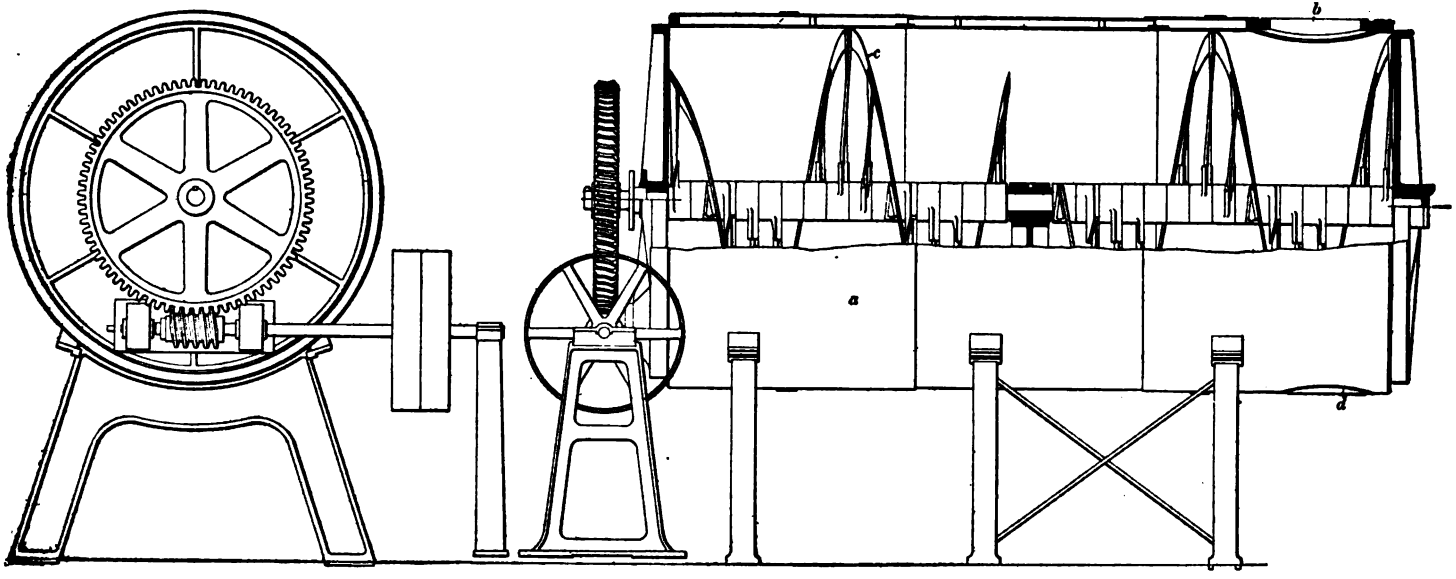


FIG. 2

the crystallization as far as possible in the vacuum pan and then continue it in the crystallizer. This is accomplished by first boiling a strike of massecuite with molasses on a crystallized nucleus, or footing, that is formed directly from sirup or from a cut of a previous grained strike and then discharging it into a crystallizer, where it is kept in motion. In another method, heavy, hot molasses is drawn into the vacuum pan on very dry massecuite without attempting to continue the crystallization in the pan; then, after thorough mixing, the diluted massecuite is discharged into a crystallizer. The crystals are kept in motion until tests of the molasses surrounding them indicate that no more sugar is being extracted.

5. Many modifications of the original crystallizer are in use. A common form of crystallizer is shown in Fig. 2. This device consists of a horizontal iron cylinder *a* provided with a spiral mixer *c*, which is driven by means of gearing, as shown. At *b* is shown an opening for charging the apparatus and at *d*, a gate, or valve, for discharging it. The massecuite is sometimes discharged through a pipe leading to the centrifugals by means of compressed air. The apparatus is usually fitted with a water-jacket *e*, which assists in regulating the temperature of the massecuite. The arrangement of the spiral mixer is such that all parts of the massecuite can be kept in motion.

In charging, the apparatus should be as warm as the massecuite, and the temperature and density of the latter should be so regulated that new crystals, or "false grain," will not form.

Occasionally, open crystallizers are used, but these are not usually considered to be so effective as the type described, because the temperature cannot be so readily regulated.

6. Following is a summary of the methods that have been used in conducting the process of crystallization in motion:

1. A strike is boiled to string-proof, that is, blank, and is run into a crystallizer containing a nucleus of grained massecuite from a previous strike. The mixture is kept

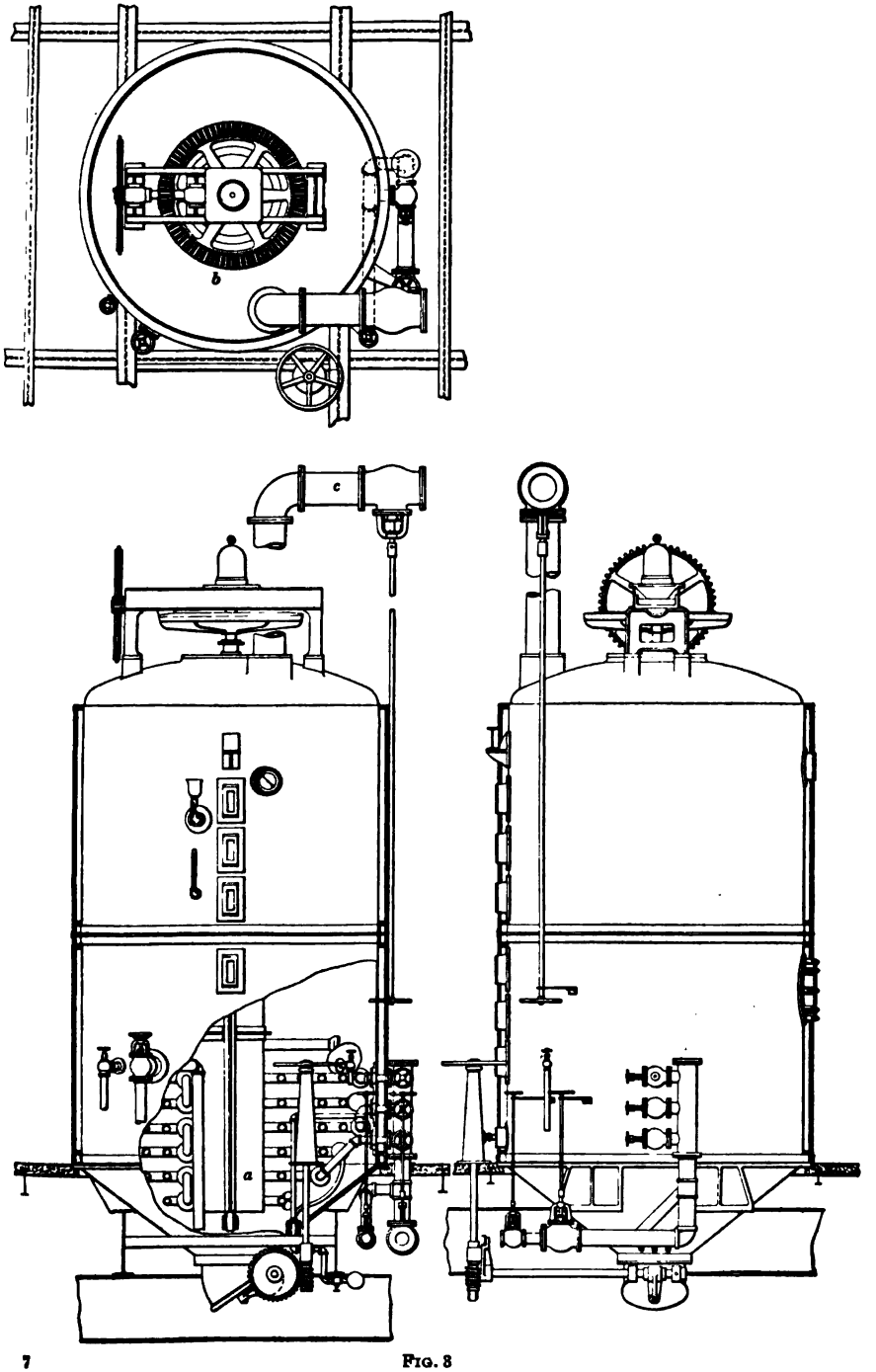


FIG. 8

in motion until the crystals no longer increase in size. (Stammer and Bock.)

2. A part of a strike of sugar is grained and boiled in a vacuum pan and is then discharged into a crystallizer containing a string strike. Molasses is added to this mixture from time to time, as it cools. (Raeymacker and Abraham.)

3. The entire strike is boiled to grain in a vacuum pan, with the boiling in of molasses in the latter part of the process. The crystallization is completed in the crystallizer. (Raeymacker and Manoury.)

4. A strike is grained with a sirup, as is customary, and is then evaporated to a very low water content; it is next diluted with from 10 to 12 per cent. of its volume of hot molasses and is discharged at a temperature of 60° to 70° C. into a crystallizer. The crystallizer is closed and connected with a vacuum pump and the air is exhausted from it. The apparatus is provided with a double bottom, or steam jacket, by means of which its contents is heated during the mixing. When the massecuite becomes very dry through evaporation, it is again diluted with 10 per cent. of its volume of molasses and the mixing and evaporation are continued. These operations are repeated until the apparatus is filled. The charging with molasses may be continuous if desired. (Hucke and Lanke.)

5. The strike is boiled in a large vacuum pan fitted with arms for mixing the massecuite during the boiling (see Fig. 3). Molasses is boiled in as in other processes. (Grossé and others.)

7. Another type of apparatus, the **Grossé vacuum crystallizer,** is shown in Fig. 3. This device resembles a vacuum pan in general construction, but differs from it in having a screw in the tube *a* driven by the gearing *b*. This screw conveys the massecuite from the lower part of the crystallizer to the upper part of the apparatus, where it falls on an umbrella-shaped deflector and is diverted back into the body of the massecuite. The object of this is to bring fresh portions of the sugar solution into contact with the crystals

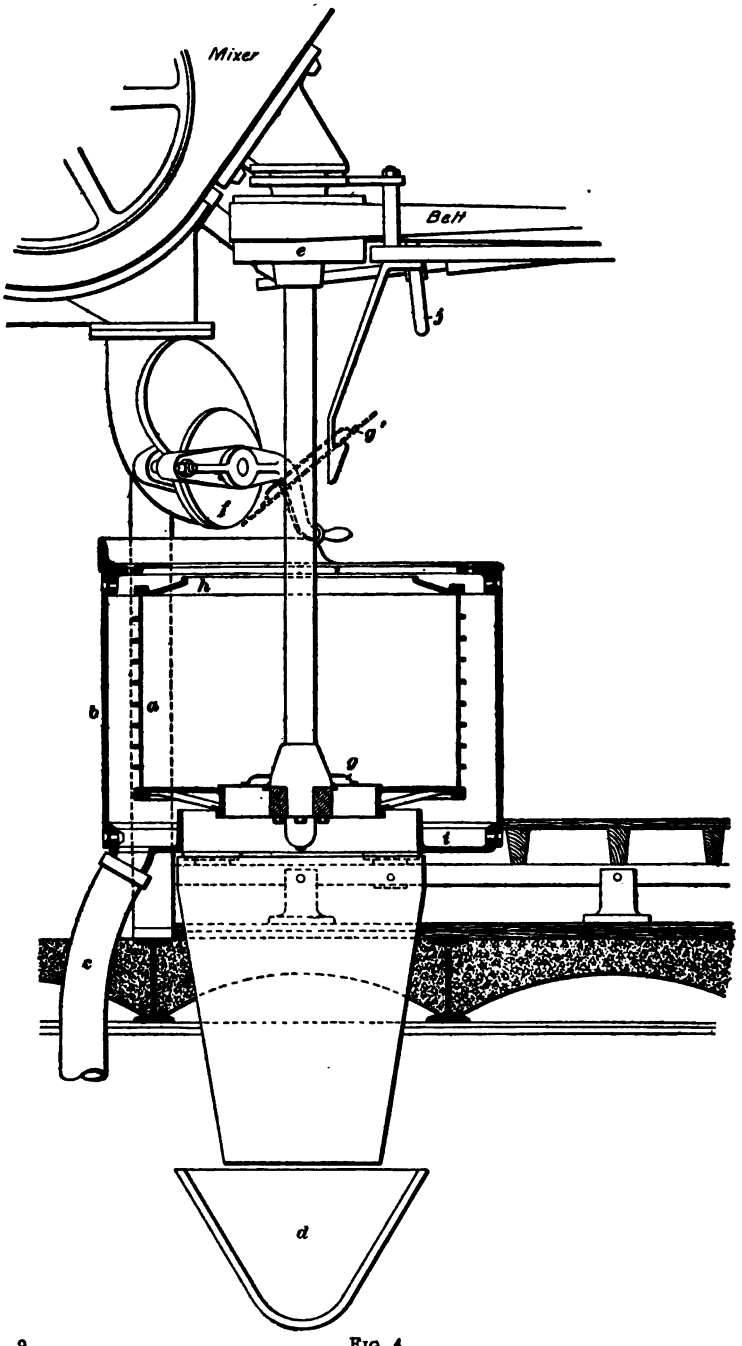


FIG. 4

and to promote the development of the latter. The vapor pipe *c* connects the crystallizer with the vacuum pump through a condenser. It is claimed that crystallization is accomplished more quickly in a vacuum-pan crystallizer of this type than in the ordinary vacuum pan supplemented by crystallization in motion.

CURING THE SUGAR

8. In curing the sugar, the massecuite is discharged directly from the vacuum pan or from a crystallizer into the mixer. It is then fed into the centrifugals from the mixer as required for purging.

A sectional view of a centrifugal and its accessories is shown in Fig. 4. The machine illustrated is of the Weston type. This centrifugal consists of a round, perforated metal basket *a* suspended by a spindle, or shaft, from a lug attached to the mixer. The usual diameters of centrifugal baskets are 30, 36, and 40 inches. At the upper end of the spindle is a bearing and pulley *e*, which is driven by a belt from a countershaft. The massecuite in the basket is prevented from overflowing by the rim *h*, which limits the size of the charge. The basket is lined with a very fine woven-wire or sheet-metal sieve. Brass or copper is used in making the lining. The basket is encircled by a stationary casing, or monitor, *b*. Centrifugals are frequently driven by water or electric motors attached directly to the head of the spindle.

9. In operating the centrifugal, the gate *f* is opened and a charge of massecuite is run into the basket, which is revolved slowly by hand or more rapidly by power, to distribute the charge. After charging, the friction clutch (not shown) that connects the machine with the driving pulley is thrown into position and the centrifugal is revolved at a rate of speed depending on the diameter of the basket. The usual speed for a 30-inch basket is from 1,200 to 1,400 revolutions per minute. The centrifugal force throws the masse-

cuite against the perforated wall of the basket *a* and carries it upwards to the rim *h*; the molasses leaves the crystals, passes through the perforations, runs down the inside of the casing into the gutter *i*, and then passes through the pipe *c* to the storage tank. If only raw sugar is being made, the operator, when he judges from the elapsed time and the appearance of the wall of sugar that the latter is dry, that is, practically free of molasses, releases the friction clutch and applies the brake *j* to stop the machine. If white sugar is being made, the operator, when he notes that the charge is practically free from molasses, sprays water on the wall of sugar from a sprinkling can, syringe, or other device while the machine is revolving. The final wash water usually contains ultramarine. The blue wash is for the purpose of avoiding the "dead" appearance, or yellowish cast, that the sugar would otherwise have. When the charge of sugar is dry, the machine is stopped and the "butterfly," or valve, *g* is lifted to the position shown by the dotted lines at *g'*. The sugar is then "cut down" and discharged through the bottom of the machine into the conveyer *d*.

10. Centrifugals are often provided with a spout *c* that can be moved from one trough to another, in order to separate the molasses from the wash sirup. This arrangement is not entirely satisfactory, as the wash sirup carries with it the molasses adhering to the casing *b* and the gutter *i*. This difficulty, however, is overcome in some machines by patented devices.

When discharged from the centrifugal, the sugar, as just mentioned, falls into a conveyer. It is then elevated to the storage bins, where, if it is to be used as raw sugar, it is ready for packing into bags or barrels. If white sugar is desired, the practice in American factories is to pass the raw sugar through a *dryer*, or *granulator*, from which it emerges as commercial granulated sugar.

11. Granulator.—The *granulator* (see *t*, Fig. 5) is an inclined, revolving iron cylinder about 25 feet in length and about 5 feet in diameter. Shallow shelves are attached

longitudinally to the inside of the cylinder, and the apparatus is so fitted that a current of hot air may be drawn through it by an exhaust fan. The sugar is conveyed into the cylinder at the elevated end and is carried part way through a revolution of the apparatus by the shelves, from which it then falls in a shower. The current of hot air from the lower end of the cylinder passes through the shower of sugar and dries it. Owing to the inclined position of the cylinder, the sugar travels through it to its lower end and there falls into revolving sieves, which remove the lumps and divide the sugar into the commercial sizes of granulated sugar. The motion imparted to the sugar in passing through the granulator separates the crystals from one another; hence, the name of the machine.

PROCESSES FOR EXTRACTING THE SUGAR FROM THE MOLASSES

12. The Molasses.—The molasses of the beet, in which the sugar cannot be crystallized, contains from one and a half to two times as much sugar as the corresponding molasses of the sugar cane. This large sugar content is due to the quantity and nature of the organic and mineral impurities in solution in the molasses. In order to recover part of or practically all the sugar remaining in the molasses, either a portion of the impurities must be removed or the sugar itself must be precipitated. The former is accomplished by the *osmose process*, which is based on the same properties of the sugar and those of the impurities as the diffusion process used in extracting the juice from the beet. In the *precipitation process*, lime and strontia are the usual precipitants.

The composition of a sample of beet molasses is shown in the following analysis:

Sucrose	50%
Raffinose	Trace
Organic matter (not sugar)	22%
Ash (mineral matter)	11%
Water	17%
Total	100%

OSMOSE PROCESS

13. The **osmose process** is an application of dialysis to beet molasses. Artificial parchment, which supplies the membrane, is held in position between the frames of an apparatus that somewhat resembles a filter press. Water is circulated in compartments formed by the parchment, on each side of which a dilute solution of the molasses also circulates. As previously described, certain bodies called *crystalloids* readily pass through a membrane, from one solution into a contiguous solution, while other bodies, known as *colloids*, do not pass the membrane at all. The crystalloids do not pass through the membrane with equal facility, certain salts passing with much greater rapidity than sucrose. If the liquids on both sides of the membrane are left in contact long enough, an equilibrium will be established as regards the crystalloids, which are present in like proportion in the two solutions. Of the crystalloids in the molasses, the salts pass the parchment membrane faster than the sugar. As it is desirable to retain the sucrose in the diluted molasses and to pass the maximum practicable proportion of the salts through the membrane, the liquids are circulated somewhat rapidly and are heated to the temperature favorable to the dialysis.

In practice, the water abstracts a part of the sucrose and about two-thirds of the saline matter. The dilute molasses from the *osmogene*, as the apparatus is often termed, is concentrated in the multiple effect and is again boiled in the vacuum pan. A part of the sugar is thus recovered by crystallization. This crystallization is made possible by the removal of the highly melassigenic salts in the osmose water that prevent the formation of crystals. It is estimated that the salts present in the molasses prevent about three and one-half times their weight of sucrose from crystallizing.

The Vallez triple osmose process is said to be especially effective and is used extensively in the American factories.

Prior to osmosis, any lime that is present should be pre-

cipitated with carbonate of soda or with acid phosphate of barium. If the solution is acid, neutral, or only slightly alkaline, caustic soda should be added to it.

At present, the osmose process is comparatively little used in Europe, owing to the demand for molasses in the distilling and cattle-feeding industries and also to the application of saccharate processes. The contrary is true in the United States, as there is little demand for molasses where the factories are located, and, besides, the osmose process is easy to apply, both in regard to machinery and to manipulations.

PRECIPITATION, OR SACCHARATE, PROCESSES

14. The **precipitation,** or **saccharate, processes** are the opposite of the osmose process, as they depend on the precipitation of the sucrose as a saccharate, thus removing it from the solution, instead of on the removal of the impurities. Several precipitation processes have been devised, but those usually applied depend on lime or strontia as the precipitant.

Calcium, Ca, forms three saccharates, or sucrates, with sucrose, of which the monobasic and tribasic are technically the most important:

Monobasic saccharate, $(C_{12}H_{22}O_{11})\cdot CaO$
Dibasic saccharate, $(C_{12}H_{22}O_{11})\cdot 2CaO$
Tribasic saccharate, $(C_{12}H_{22}O_{11})\cdot 3CaO$

The monobasic saccharate is soluble in water and insoluble in alcohol. On heating the water solution to boiling, all the lime combines with one-third of the sucrose to form the tribasic saccharate, which is not readily soluble in water. The dibasic saccharate is soluble in warm water, but in cold water, it separates in the crystalline form.

If 3 molecules of lime is added to the sugar solution, the temperature being kept below 30° C., the tribasic saccharate will be precipitated. As will be noted later in the description of the Steffen separation process, the temperature of the solution has a marked influence on the physical condition

of the precipitate. This saccharate is soluble with difficulty in 200 parts of water and is insoluble in a solution of calcium hydrate.

15. *Strontium*, *barium*, and *lead* also form saccharates with sucrose, but not so many as the lime.

Strontium forms two saccharates—the monobasic and the dibasic. The monobasic is formed in the cold, while the dibasic is formed at 100° C. or more readily at a higher temperature. This saccharate is not very stable.

Barium forms one saccharate—the monobasic. This is not used in sugar manufacture owing to the scarcity of the reagent.

Lead forms three saccharates, of which, one, the tribasic, has been used to some extent in sugar manufacture.

Lead and barium are usually considered objectionable on account of their toxic properties.

While saccharates formed with each of the bases mentioned have been used, those of the greatest commercial importance are the tribasic saccharate of lime and the dibasic saccharate of strontium. The selection of the base depends largely on local conditions as to the ease of obtaining a supply of the material. The German factories use both lime and strontium processes, and the French and American factories, which cannot readily obtain strontia, use Steffen's tribasic saccharate-of-lime process, the separation modification.

In order that a clear idea of the development of these precipitation processes may be formed, methods that are no longer employed, as well as those in current use, will be described.

16. Elution Processes.—The original **elution process**, which is one of the oldest precipitation processes, was invented by Lair and Bilange, in France. In this process, the sugar is precipitated as a tribasic saccharate of lime. On the addition of powdered quicklime to the molasses, the saccharate immediately forms and the mass solidifies. The solid mass is broken into small fragments and is then extracted with water so as to remove the impurities. The

saccharate either is used in liming the juice, preparatory to the carbonatation, or is heated nearly to the boiling point in water and is carbonated and subsequently treated in the same manner as juices.

Scheibler modified the process and gave it the name "elution." In this modification, the tribasic saccharate is obtained as in the original method, except that lime that has been recently slaked is used. After drying the saccharate on hot plates, it is broken into small pieces, which are systematically washed with 37- to 40-per-cent. alcohol. The saccharate is insoluble in the alcohol, but most of the impurities are soluble in the reagent and may be removed by it. The alcohol is drained from the saccharate, the last traces of it being removed by steaming, and it is recovered by distillation. The saccharate is further treated as already described for the recovery of the sugar.

17. Seyferth further modified the process by thoroughly mixing 44° Baumé molasses with 30 to 40 per cent. of its weight of finely powdered quicklime and then transferring the mixture to small iron tanks, which he only partly filled, for the completion of the reaction. The mass heats to about 125° C. and increases greatly in bulk, and the water evaporates from it. Care must be taken to prevent the mass from overheating locally and taking fire through imperfect mixing of the lime and molasses. After removing the impure saccharate from the tanks and cooling it, it is broken into small pieces and washed with alcohol.

There is danger of the sugar becoming decomposed in applying both this modification of the process and that of Scheibler, on account of the heat that is generated when the lime combines with the sugar. In order to overcome this defect, Manoury further modified the elution process as follows:

The molasses is reduced to 42° Baumé, and carbonate of sodium is added to it, so as to break up the lime salts of the organic acids, which are insoluble in alcohol. The prepared molasses is next mixed with powdered slaked lime in a

special apparatus, a sufficient quantity being used to precipitate all the sugar as the tribasic saccharate. The slaked lime is prepared by dipping quicklime into water and then placing it on a floor to complete the slaking; the resulting powder is sifted and the molasses is added to it. The mixer produces a granular saccharate and this is washed systematically with alcohol, the latter remaining in contact with the saccharate about 20 hours. After the washing, the last of the alcohol is expelled from the saccharate with steam, which also reduces the material to the consistency of a milk, which is used in liming the juice preparatory to carbonating.

This elution process is the only one that has been generally applied. Owing to the impurities extracted in washing the saccharate, the alcohol that is used cannot be readily redistilled.

18. Steffen's Substitution Process.—If the monosaccharate of lime in water solution is heated to boiling, all the lime will combine with one-third of the sugar and form the tribasic saccharate, which is not readily soluble in water. The remaining two-thirds of the sugar is set free. These properties of the saccharates were utilized by Steffen, an Austrian engineer, in his "substitution" process, which is of importance as the forerunner of his "separation" process, now widely applied in the American beet-sugar factories.

The molasses is diluted to a sugar content of about 10 per cent., and then sufficient lime is added (28 per cent. of the sugar) to form a monosaccharate with the sugar. After agitating the mixture for 8 to 10 hours at a temperature of about 10° C., it is transferred to closed vessels, where it is heated, under pressure, to a temperature of 110° C. The tribasic saccharate of lime, which separates at this temperature, is removed by filtration through presses, and is then suspended in 120 per cent. of its weight of boiling water and again filter-pressed; the cake is washed with 35 per cent. of boiling water. The tribasic saccharate is utilized as has been previously described. To the mother liquor and the filtrates from the second filter pressing is added sufficient

molasses to restore the sugar content to 10 per cent. of the solution. In other words, the sugar of a fresh portion of molasses is substituted for that removed in the tribasic saccharate; hence the name **substitution process.** This diluted molasses is treated as before, and one-third of the sugar it contains is removed in the saccharate. As a rule, these manipulations are repeated about twenty times, fresh molasses equivalent to the sugar removed being substituted each time, until the mother liquor becomes so impure that it can be treated only with difficulty. At this stage of the process, the sugar, with the exception of a small quantity, is removed by repeated precipitations without the addition of molasses.

This process has been superseded by the *Steffen separation process.*

19. Steffen's Separation Process.—**The Steffen separation process** is used by several American beet-sugar factories and yields very satisfactory results. This process depends on the precipitation of all the sugar in the molasses as the tribasic saccharate of lime, under temperature conditions that produce a very granular precipitate. The precipitant is freshly burned quicklime that has been recently reduced to an impalpable powder and stored out of contact with the air.

20. The process is conducted as follows: A quantity of molasses is reduced with water to a density of 12° Brix, and to a percentage of sugar of about 7. The solution is placed in a closed mixer provided with cooling tubes and is cooled to a temperature of 15° C. Small, measured portions of lime are then added at intervals of about 1 minute. At this temperature, the lime combines with the sugar in preference to the water, and therefore does not hydrate. With each addition of lime, a rise of temperature is brought about by the chemical combination of the lime and the sugar. Each time, before adding a portion of lime, the temperature of the liquid must be reduced to below 18° C. The addition of lime is continued until a test sample shows that all the sugar

has been precipitated. In making this test, it is customary to filter the test sample through paper and then to take the specific gravity of the filtrate. The Brix hydrometer is used for this purpose, and should read in the filtrate, from 6° to 6½° when the precipitation is completed or, preferably, until the density no longer changes with the addition of lime. In the early work with this process, from 130 to 140 pounds of lime was used to precipitate the sugar from 100 pounds of molasses, but this quantity has been reduced to less than 100 pounds.

21. The tribasic saccharate produced at low temperatures under the conditions described is very granular and may be readily separated from the mother liquor by filter pressing under low pressure. The crude saccharate suspended in the mother liquor resembles dirty milk of lime. The mother liquor is removed by filtration, and as it contains the potash salts of the molasses, it may be used as a fertilizer, or the potash may be recovered. The saccharate is washed in the filter press with cold water, and the wash water, which contains saccharate of lime in solution, is used in diluting a fresh lot of molasses.

The washed precipitate contains from 95 to 98.5 per cent. of pure tribasic saccharate of lime, and is used in liming the hot juice for the carbonatation. The saccharate may be reduced to a thin milk with hot water and the lime precipitated with carbonic acid, leaving a very pure sugar solution. It is necessary to use very hot juice or hot water in reducing the saccharate to a milk, since in the cold, the carbonic acid causes the granules to become coated with carbonate of lime, which occludes particles of the undecomposed saccharate and results in a loss of sugar.

22. The quantity of lime in the saccharate is usually greater than the requirements of the carbonatation process, especially if the plant treats molasses from other factories in addition to its own. In this event, a part of the saccharate is mixed with hot juice and the hot mixture is filter-pressed. A soluble saccharate and slaked lime are formed under the

influence of the heat and the sucrose in the juice. The slaked lime, together with the excess of lime, used in preparing the tribasic saccharate, is retained by the press, and the lime combined with the sugar is utilized in the carbonatation process.

The Steffen separation process requires the installation of a refrigerating plant, except when used during the winter months in cold climates.

With a view to the economy of lime, Bauer devised and patented a continuous rotary kiln for calcining the lime cake from the carbonatation. The lime produced is again utilized in the saccharate process. This type of kiln is used in several American factories.

STRONTIUM PROCESSES

23. Two methods devised by the eminent sugar chemist, Doctor Scheibler, have been extensively used in Germany. These are known as the *strontium dibasic* and *strontium monobasic processes*. The dibasic process was patented in 1880 and the monobasic in 1882.

24. Dibasic Strontium Process.—The hydrate of strontium, used as a precipitant in the **dibasic strontium process,** is obtained from the mineral strontianite—carbonate of strontium. The strontianite is calcined, and the resulting oxide is converted into the hydrate by treatment with hot water. The hydrate dissolves, and the solution is separated from the impurities by decantation and crystallization. After the factory is once supplied with strontium hydrate, only a small quantity of strontianite has to be calcined from time to time to compensate for the losses in the manufacture.

The hydrate of strontium in solution and the molasses are heated to the boiling point and mixed together in a tank provided with a steam coil or a blow-up pipe. The strontium is added in excess of the quantity required to produce the dibasic saccharate. The mixture is boiled, and saccharate separates in a granular form. The saccharate and the mother liquor are thrown on a metallic screen, and the liquor is

drawn off by suction. The precipitate is washed with a hot 10-per-cent. solution of strontium hydrate, and the washings are used in preparing another quantity of the saccharate. The mother liquor is cooled, and the excess of the hydrate is recovered from it by crystallization. The saccharate is transferred to a series of communicating tanks, and water cooled to below 15° C. and containing 2 per cent. of strontium hydrate is passed through the saccharate in the first tank, thence to the second tank, and so on. During this operation, cold water circulates about the tanks to cool their contents. At this stage of the process, the saccharate decomposes, the sugar and a part of the strontium passing into solution and the strontium hydrate crystallizing. The hydrate so formed is used in treating a fresh quantity of molasses. The sugar solution is carbonated, and the strontium carbonate is separated in filter presses and recalcined and treated as described for the strontianite. In lieu of the separation of a part of the strontium hydrate from the sugar by crystallization, it may all be precipitated by carbonic acid. The sugar solution is utilized as at other stages of the manufacture.

25. Monobasic Strontium Process.—In the **monobasic strontium process,** the molasses is added little by little to a hot solution of strontium hydrate obtained as described in connection with the dibasic process and containing 25 per cent. of the hydrate. The temperature falls with the formation of the monosaccharate and must be further reduced to 20° C. by cooling. The strontium hydrate must be used in excess of the quantity required to form the monosaccharate. The precipitate is separated from the mother liquor by filter pressing and is washed with cold water, after which it is suspended in water and the strontium is precipitated as a carbonate. The carbonate of strontium is removed from the solution by filter pressing. After washing the carbonate, it is molded into small blocks and then calcined and converted into the hydrate, as already described.

The washings from the saccharate, which contain some sugar, are treated with strontium hydrate and then boiled to

precipitate the dibasic saccharate; this is separated by hot filtration, and then used with strontium hydrate in treating fresh molasses. The strontium is separated from the filtrate by crystallization and carbonatation.

The solution of sugar obtained on carbonating the monosaccharate of strontium is either treated separately in the factory or mixed with the carbonated juice, according to whether the work is being conducted during the crop or in the dead season.

MISCELLANEOUS MOLASSES PROCESSES

26. Many modifications of the molasses processes described have been devised and abandoned; also, other processes have had a very limited application. The latter processes include precipitation of the sugar as barium saccharate and as lead saccharate.

27. Remarks on the Molasses Processes.—The osmose and the Steffen separation are the only processes used in America. Owing to the abundant deposits of strontianite in Germany, the strontium process is extensively employed in that country, but it is seldom used elsewhere. Table I shows the relative importance of the molasses processes in Germany. These figures show percentages computed from German statistics. For many years the quantity of molasses treated per annum has varied little in Germany.

TABLE I

COMPARATIVE STATISTICS OF THE USE OF MOLASSES PROCESSES IN GERMANY

Crop	Osmose Process Per Cent.	Steffen's Separation Process Per Cent.	Strontium Process Per Cent.	Elution Process Per Cent.	All Other Processes Per Cent.
1889–90	16.5	14.3	41.9	24.9	2.4
1894–95	6.4	13.4	70.2	10.0	None
1898–99	1.5	7.3	89.0	1.3	0.9

UTILIZATION OF BY-PRODUCTS

28. Exhausted Cossettes, or Pulp.—In Europe, the by-product of beet-sugar manufacture, namely, the **exhausted cossettes,** or **pulp,** is fed to cattle and sheep, but at some American factories it is often wasted. A few plants, however, have found a market for the moist pulp by selling it as cattle food. For this purpose, the pulp is conveyed from the diffusion battery to continuous presses of the Klusemann, Bergreen, or other type, and a part of the water it contains is removed from it.

The *Klusemann press* is a device for forcing the pulp through a perforated iron cylinder, in which is placed a cone-shaped shaft that is fitted with steel blades arranged in the form of a helix. The conical shaft is also perforated. The cylinder has a jacket that leads the water pressed from the pulp to outlet pipes at the bottom; a pipe also conducts the water from the inside of the cone. As the pulp descends in the cylinder, the space it may occupy becomes smaller and smaller and the consequent pressure forces the water from it. The wet pulp is fed continuously into the cylinder at the top, and the pressed pulp is discharged at the bottom. The wet pulp contains about 95 per cent. of water and the pressed pulp, 87 per cent. The efficiency of the pressing depends somewhat on the quality of the beets and the working conditions in the diffusion battery. Thin cossettes from sound beets that are extracted without excessive heat and delivered to the presses at a temperature of about 60° C., are readily pressed.

29. The wet cossettes are often stored in large piles on the ground near the factory, drainage ditches being provided to carry off the water. The upper surface rots, but the lower layers keep fairly well and are greedily eaten by cattle. This method of storing, however, results in a great loss of nutritive matter. The pulp is often stored in cemented excavations, or silos, that have a covering of straw and earth. In such cases, arrangements must also be made for the removal of the drainage water. In recent years, much attention has been

given to the drying of beet pulps for shipment. The dry pulp is used as cattle food and as an absorbent for a liquid food, such as molasses or blood or a mixture of these. There are several kilns in use for drying the cossettes, but a description of these will not be necessary for the present purpose. The temperature of the kiln may be high, but owing to the rapid evaporation, that of the pulp does not reach 100° C. The dried pulp contains from about 12 to 15 per cent. of moisture. It is not necessary to reduce the water content below these figures, since the dried pulp will immediately reabsorb this quantity on exposure to the air. The dried-pulp or molasses-pulp foods prepared in this manner may be shipped in bags, and under proper storage conditions they will keep indefinitely.

30. Molasses.—In the factories that do not use a saccharate process, there is a large molasses residue. This is utilized either by mixing it with dried beet pulp for use as cattle food or by fermenting and distilling it, with the subsequent recovery of the potash salts from the final residue, which is termed *vinasse*.

The molasses is prepared for distillation by dilution and fermentation. A very small proportion of sulphuric acid is often added to the diluted beet molasses in order to promote the vinous fermentation and to check objectionable ferments. After distilling off the alcohol from the fermented molasses, the vinasse is evaporated to dryness in multiple-effect evaporators or in large iron pans and the residue is then ignited. The organic matter burns off, leaving a black ash that is very rich in potash salts. The salts are recovered by lixiviation, followed by concentration and crystallization.

In this process, as usually applied, all the nitrogen is lost. It has been proposed by a French chemist, M. Vasseux, to concentrate the vinasse to a certain density determined by experience and then to add sufficient sulphuric acid to it to convert practically all the potash salts present into a sulphate. Under suitable conditions of concentration, nearly all the potash sulphate crystallizes and it is removed by means of a centrifugal. The mother liquor is concentrated and used

as a nitrogenous fertilizer. The nitrogen may also be saved by submitting the dried vinasse to dry distillation in a retort.

A very porous charcoal, a liquid distillate, and gases are the products of the distillation. The potash is recovered from the charcoal by lixiviation and crystallization, and the gas is used for heating purposes. Besides a small quantity of tar, the liquid portion contains ammonium carbonate, sulphohydrate and cyanide, methyl alcohol, trimethylamine, and volatile fatty acids. By a somewhat complicated process, methyl alcohol, ammonium chloride, trimethylamine, and methyl chloride are obtained. The methyl chloride when compressed to a liquid is used in the manufacture of aniline dyes. This method of treating the vinasse economizes a very large part of its nitrogen.

The molasses from the beet is not usually suitable as food for human beings. Both its taste and its odor are very disagreeable. Several processes have been devised, notably in Germany, for removing or disguising the disagreeable properties of the molasses so as to make it fit for table use.

ARRANGEMENT OF MACHINERY IN A BEET-SUGAR FACTORY

31. Fig. 5 illustrates a section of the Los Alamitos sugar factory, erected in 1897 by E. H. Dyer and Company. Referring to this illustration, *a* shows the beet elevator; *b*, the beet cutter from which the cossettes are delivered into the diffusion battery *c*; *d*, a receiving tank; *e*, the first carbonators; *f*, the second carbonators; *g*, the filter presses; *h*, the engine that drives the beet slicers and elevators; *i*, the engine that drives the centrifugals and crystallizers; *j*, the water tank, to supply the diffusion battery; *k*, the strike, or vacuum, pan; *l*, the air pump, to remove air and gases from the strike pan; *m*, the tank, to hold the sirup preparatory to being boiled in the strike pan; *n*, the molasses tank; *o*, the crystallizers; *p*, the mixer; *q*, the centrifugals; *r*, the conveyer that carries the sugar from the centrifugals and delivers it into the elevator *s*; *t*, the granulator, or

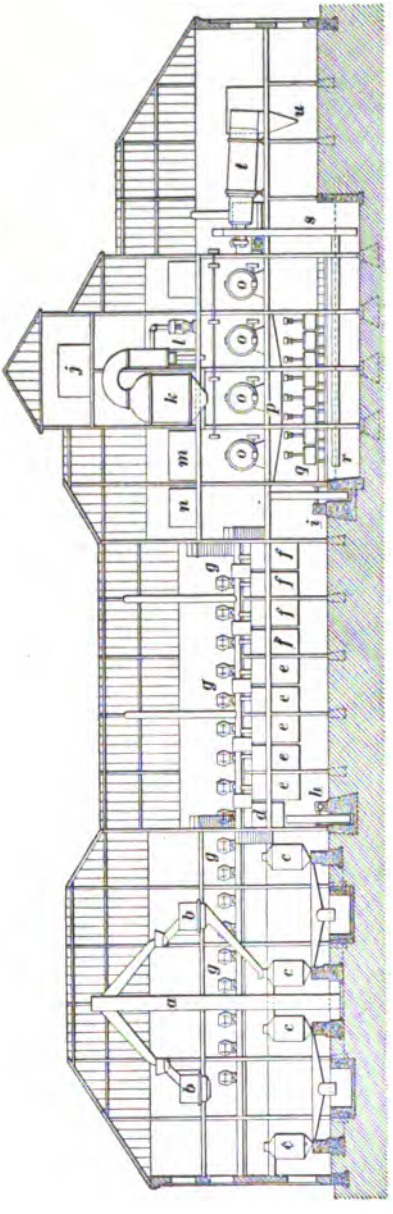


FIG. 5

sugar drier; and *u* the hopper, from which the sugar is packed for shipment.

32. Figs. 6 and 7 are illustrations showing the main floor of a sugar house. At *a*, Fig. 6, is shown the elevator that conveys the beets from the washer to the slicer *b*, where they are cut into cossettes, and at *c*, the chute through which the diffusers of the battery *d* are charged with cossettes. The exhausted cossettes are dropped into sluices, or conveyers, below the battery, where they are loaded into wagons, and then taken to the silos, or storage place, to serve as food for cattle. The diffusion juice is measured in the tanks *e* and then flows into the carbonatation tanks, or carbonators, *f*. After the liming, using milk of lime from the measuring tanks *i*, and the carbonatation, the juice is filtered through the

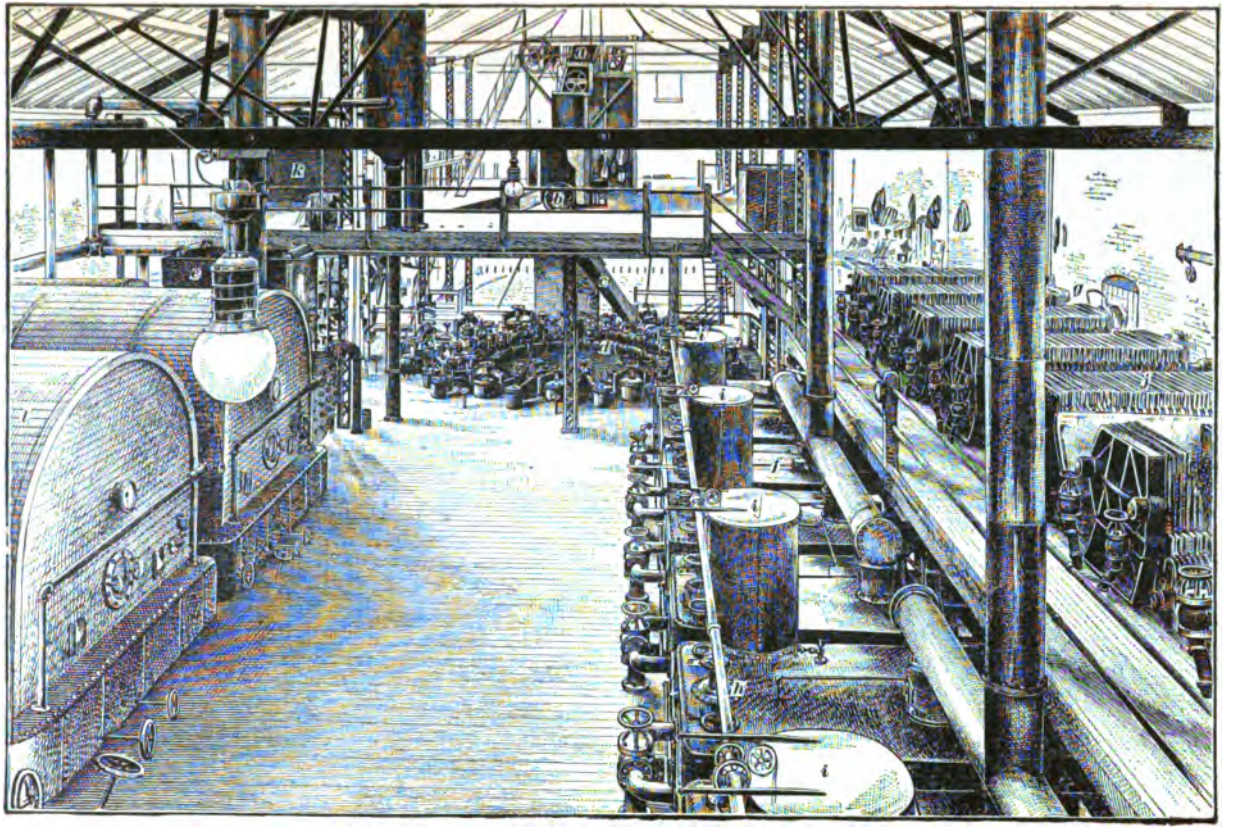


FIG. 6

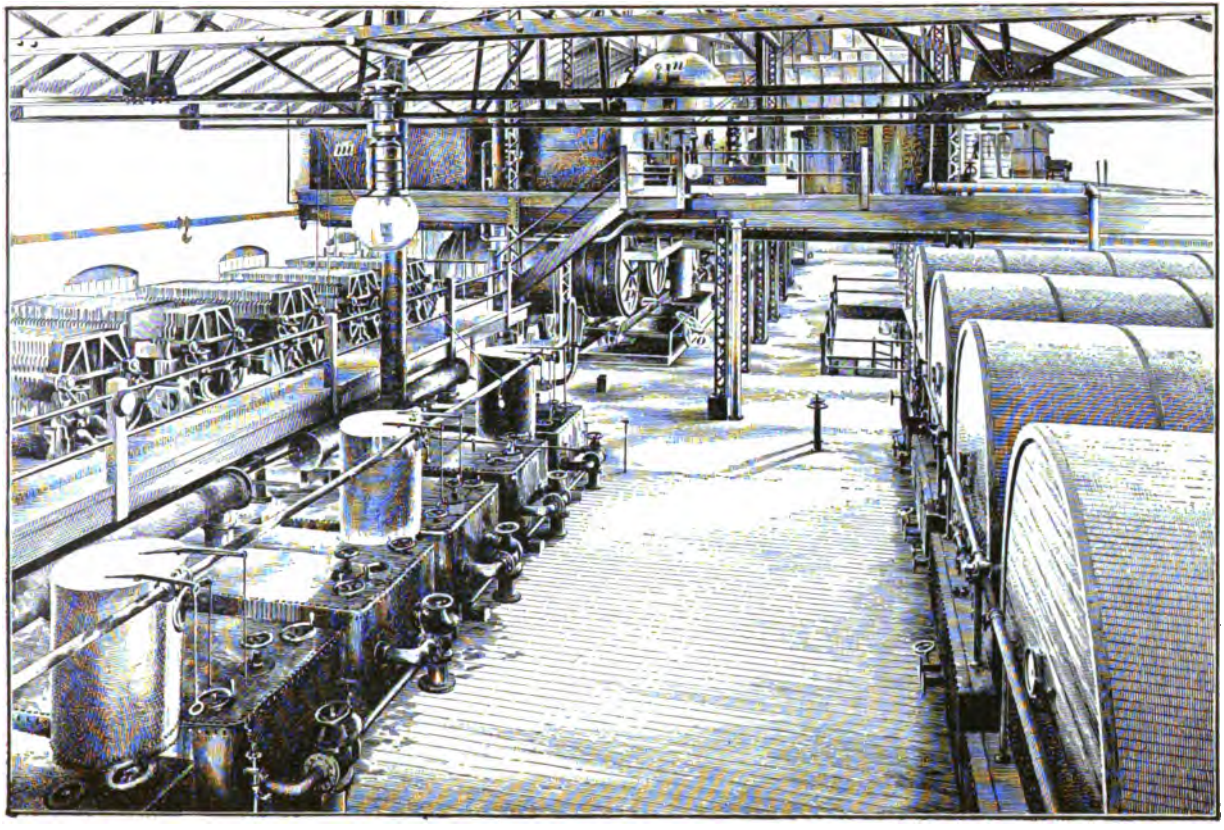


FIG. 7

presses *g*, and the filtrate is conveyed to the saturation, or second-carbonatation, tanks, *h*. When the saturation is completed, the juice is again filter-pressed, this time in the presses *j*, and the filtrate is pumped to the sulphuring tanks *k*. After sulphuring, the juice is filtered through mechanical filters, not shown in the figure, and the filtrate is concentrated in the quadruple-effect evaporator *l*. The sirup from the evaporator is sulphured and passed through mechanical filters, not shown, and is stored in the charge tank *m*, Fig. 7, ready to be boiled to grain in the vacuum pan *n*. The massecuite is conveyed from the vacuum pan to the mixer *o* of the centrifugal machines in order to be purged, or first to the crystallizers *p*, according to the method of manufacture.

REFINING OF SUGAR

INTRODUCTION

33. The refining of raw sugars is conducted on a very large scale in many of the seaboard cities of the United States. With few exceptions, the factories of Louisiana and Texas produce raw sugars, though at one time almost the entire product of these states was suitable for immediate consumption. From the introduction of the industry, the American beet-sugar factories have, with only two or three exceptions, manufactured a high grade of granulated sugar, ready for consumption. As a rule, the tropical countries produce sugar suitable only for refining.

34. Many grades of raw sugars are imported for the American refineries, certain establishments preferring and working certain grades for which they are best equipped. Table II, in which are given the analyses of a few typical grades of raw sugars, has been prepared from data obtained by Dr. W. D. Horne, chemist of the National Sugar Refining Company and supplied through his courtesy.

TABLE II

ANALYSES OF RAW SUGARS

Country	Grade	Per Cent. Sucrose (Polarization)	Per Cent. Ash	Per Cent. Water	Per Cent. Glucose	Per Cent. Organic (Non-Sugar)
Cuba	Centrifugals	95.30	0.66	1.55	1.10	1.39
Cuba	Muscovado	88.53	1.20	4.43	3.05	2.79
Cuba	Molasses sugar	88.25	1.66	3.88	2.70	3.51
Java	Centrifugals	97.60	0.41	0.53	0.82	0.64
Mexico	Centrifugals	94.67	0.66	1.48	1.70	1.49
Mexico	Muscovado	89.39	1.33	3.50	2.47	3.31
Mexico	Molasses	89.32	1.43	3.45	2.64	3.16
Macoris . . .	Concrete	83.15	2.42	4.52	3.68	6.23
Macoris . . .	Centrifugals	96.27	0.48	1.05	0.99	1.21
Macoris . . .	Muscovado	92.20	0.86	3.75	1.61	1.58
Macoris . . .	Molasses sugar	86.71	2.53	4.08	1.86	4.82
Germany . . .	Beet sugar	94.57	1.01	2.26		2.16
Austria	Beet sugar	94.74	1.12	1.80		2.34
Hungary . . .	Beet sugar	94.57	1.05	2.21		2.17

This list given in the table does not include some of the very low-grade sugars. The grade known in the market as *centrifugal* is produced from sugar cane and is of the first crystallization in a vacuum pan. The modern raw cane-sugar factory produces little except the centrifugal sugar. The *muscovado* is made from cane, but the sugar is grained in coolers from liquor that is concentrated in an open pan, or kettle. *Molasses sugar* is made from cane molasses and is obtained from strikes boiled "blank" or to "string-proof." A similar sugar, called *beet seconds*, is made from beet molasses. The grade known as *concrete* is obtained by concentrating cane juice until it solidifies on cooling. The evaporation is usually conducted in open vessels. Concrete therefore contains all the impurities of the clarified juice. The sugar that is usually called *beets* by the refiners is the first crystallization of beet sirup.

The centrifugal sugar is the best refining grade. This is followed by beets and muscovado. Molasses sugar, on account of its soft grain and high percentage of impurities, grades below the muscovado. The expense of refining concrete sugar is very high, since the impurities usually eliminated in the factory are brought with it to the refinery.

35. Raw cane sugars are sold to the refineries on a basis of their polarization and grade. There is an arbitrary deduction made from the polarization of beet sugar to determine the "titrage," or "test," on which the price is based. This deduction is generally five times the percentage of ash in the sugar. On the importation of raw sugar, the duty is based on the polarization of the sugar, together with its color, as referred to a set of colors prepared in Holland and called the *Dutch standards*.

Ninety-six degrees, polariscopic test, is the usual standard for centrifugal sugar. A deduction is made for each degree that the test falls below 96° and additional is paid per degree above this number, namely, $\frac{1}{16}$ cent per degree above and $\frac{1}{8}$ cent per degree below 96°. This scale is made on the assumption that sugars below the 96° test are more difficult to refine than those of 96° or higher. Muscovado and molasses sugars are sold on a basis of 89° test.

As already indicated, the method of refining depends largely on the grade of the raw sugar. The process used for the highest refining grade, centrifugal sugar, will now be considered.

SUGAR-REFINING PROCESS

36. The centrifugal sugar is dumped into an elevating mixer, called a *magma machine*, where it is mixed with sirup so as to form a magma of about the consistency of massecuite. As fast as the magma is formed, it is elevated and discharged into the mixer of the centrifugal machines. The sirup softens the coating of impurities that is on the crystals and prepares the latter for washing in the centrifugals. As is customary with massecuites, the magma is purged in the

centrifugals, and the crystals are thoroughly washed with water. The resulting sugar is of a very light straw color and of a polariscopic test about 99°. This sugar is next dissolved in the melters, forming a solution containing about 50 per cent. or more of sugar, and is then pumped to steam blow-up tanks. The sirup obtained in washing the raw sugar is pumped to separate blow-ups for treatment apart from the melted sugar, since the sirup contains most of the impurities of the raw material. This method enables the refiner to work 89° "test" beet sugar with cane sugar. The washed sugars are mixed, and the wash sirup containing most of the impurities is mixed with other low-grade materials.

The melted sugar is limed to slight alkalinity in the blow-up tanks, and then sufficient phosphoric acid is added to combine with the lime. After thoroughly blowing up the liquor with steam and skimming it, it is filtered through bag filters.

37. Taylor Filters.—The filtering apparatus used in sugar refining is known as the **Taylor filter.** The bags of this filter are about 5 or 6 feet long and from 4 to 6 inches in diameter. These bags are made of strong, twilled cotton cloth, and each is slipped into a slightly longer outer bag, or sheath, made of strong coarse cloth. The mouth of each filtering element is tied over a nozzle, which is then screwed into a suitable hole in an iron plate. This plate forms the bottom of a very shallow tank and the top of the filter box, or chamber. The filter box is usually of sufficient size to accommodate a large number of filter bags, and is provided with suitable doors to give access to the interior; it also has steam connections and pipes for filtered liquor.

During filtration, a very shallow depth of liquor is maintained in the feed-tank. The bags gradually fill with the lime precipitate and the impurities that are separated during the treatment of the liquor in the blow-up. At this stage, the mud in the filter bag is washed with hot water, while steam is admitted into the filter box to maintain the temperature. The wash water is utilized in melting sugar or in

some other process, so that none of the sugar it contains will be lost. The mud is washed from the bags and then filter-pressed. The filtrate and the water from washing the bags are concentrated in order to recover the sugar they contain. It should be noted that in the case of the Taylor, or bag, filter the filtration takes place inside of the bag. This is just opposite to the way that it is carried on in the mechanical filters used in sugar factories.

The sirup molasses drained from the raw-sugar crystals in purging the magma, as previously described, is also treated the same as the melted sugar, but on account of its low purity, it is kept apart from the latter at all except the last stages of the process.

38. Char Filters.—From the bag filters, the liquor is conveyed to the **char filters,** which are large, vertical iron cylinders with perforated false bottoms. There is also a tight-fitting door, or cover, at the top of the filter and a discharge door for char at the bottom. Pipes and valves are arranged to conduct the liquor into any filter and to draw off the filtrate. The filtration is conducted under pressure, and the discharge pipes for the filtered solutions terminate above the filters in plain view of the attendant. The filtering medium is animal charcoal prepared from bones. This material, in small fragments, is packed on blankets, which are laid on the false bottoms of the filters. The strained sugar solution from the bag filters is run into the char filter until it displaces the air and completely fills the filter. After a certain length of time, determined by experience, the discharge and inlet valves are opened and a continuous stream of sirup, or final molasses by-product, is passed through the filter from top to bottom. The pipes discharge into a conveniently arranged trough near the top of the filter. For sometime the liquor flowing from the filter is water white and clear; it then has a faint tinge of yellow, and, finally, when this color deepens, water for washing the char is substituted for the liquor. The washing is continued until tests show that the water contains too little sugar to justify saving

it. The wash waters are concentrated to heavy liquors in multiple-effect evaporators. The sirup is graded according to its color.

During the filtration, the char absorbs certain organic and inorganic impurities and a small quantity of sugar from the sirup and decolorizes the latter. The filtered liquors are boiled in vacuum pans for the crystallization of the sugar, as in the factory processes previously described. The molasses from the sugars is boiled in just as long as its color will permit without affecting the quality of the refined sugar, or it is used in making soft, yellow sugars. The sirup obtained in the first stages of the process of refining is treated precisely the same as the liquor from the melted sugar, and when the molasses from this sirup becomes too highly colored, it is mixed with the same grade from the melted sugar, to produce raw sugar. The very low grades are finally treated in the crystallizers, as in the factory methods.

Molasses sugars are often mixed with centrifugal sugar at the factories and also at the refineries, and are then treated as described for centrifugal sugar. Certain very low-grade sugars are melted, and the resulting liquors are worked separately from the high-grade sugars. The sugars are purged in the centrifugals and are washed and dried, or granulated, as in the factory methods.

39. The product known as **cube sugar** is made by moistening soft, white sugar with white sirup and molding it in a special machine. The cubes are dried in an oven. **Loaf sugar** is cut or partly sawed and broken from molded slabs, or loaves. **Powdered sugar** is produced by grinding or crushing the crystals.

The factories of continental Europe use sulphurous acid to some extent for bleaching the liquors, so as to economize bone black, and depend on systematic washing of the sugar with sirup to remove the molasses.

40. Revivification of the Char.—The bone black, or char, used in decolorizing the liquors soon ceases to be effective through the absorption of organic and mineral

matters. The char is not rejected, however, but is revivified by washing and reburning.

The washing is effected in the filter by passing boiling water through the char. In European practice, where raw beet sugar is almost exclusively used for refining, the char is sometimes washed with dilute hydrochloric acid also, so as to remove the carbonate of lime. After thorough washing with water, the char is conveyed from the filters to the kilns for reburning. The conveyer deposits the char on top of the kiln for a preliminary drying.

The char kiln used by refineries usually consists of a number of small, vertical, cast-iron pipes, about which circulate the heated gases from a furnace. The upper ends of the pipes terminate in a storage or drying platform for the wet char, and the lower ends, in sheet-iron cooling pipes. The maximum temperature maintained in the tubes is about 650° C. The apparatus is so arranged that air will not enter the hot zone of pipes of the kiln.

The wet or partly dried char enters the upper parts of the pipes and gradually works its way downwards to the cooling pipes as charges are withdrawn. These pipes are provided with valves, or gates. As the char descends, it passes through the highly heated zone of the pipes and the organic matter is carbonized; from the hot zone, it passes into the cooling pipes and is drawn off for use from time to time.

The color of the revivified char is indicative of the efficacy of the treatment. A reddish char is underburned; a gray char shows that air has leaked into the pipes and part of the carbon has been consumed; a white char is overburned; a brilliant black char indicates proper burning.

On account of the absorption of mineral matter, the weight of the bone black increases with use. According to Gallois and Dupont, bone black of good quality that has been used should not weigh more than 1.23 times its weight when new. A gain of 47 per cent. in weight indicates that its condition is bad, and one of 50 per cent. that it should be rejected.

Refiners use a part of the spent bone black in manufacturing phosphoric acid, or superphosphate of lime, which is

used for precipitating the lime when blowing up the liquors, as already described. The excess of spent bone black is utilized in the manufacture of phosphatic fertilizers.

41. By-Products in Refining of Sugar.—The only by-products in the refining of sugars is the *final molasses*, or *barrel sirup*. This material is often treated for the improvement of its color and is then utilized in the production of table sirup. It often reaches the hands of dealers that use it for mixing with starch sirup or glucose for table and bakers' use. Large quantities of the molasses are utilized in the manufacture of alcohol.

COTTONSEED OIL AND PRODUCTS
(PART 1)

HISTORY AND DEVELOPMENT OF COTTONSEED-OIL MANUFACTURE

PRODUCTION OF COTTON

1. Origin of Cotton.—The origin of the cotton plant has never been determined definitely. Herodotus, the Greek historian, in his writings about 450 B. C., refers to the cotton fiber as "tree wool" and says that the people of India had long been accustomed to use it for making their garments. He writes of trees in India bearing as their fruit fleeces more delicate and beautiful than those of sheep. Frequent references to cotton in the "Sacred Institutes of Manu" show that the fiber was in use at the time when those ancient laws were compiled, about 800 B. C. From early references to cotton in India arose the belief that it originated in that country and spread thence into other countries. Whether this view is correct, or whether cotton is indigenous to several countries, is an unsolved problem. After India, China appears to have the oldest claim, and Persia probably comes next. Rhinds, however, has advanced the theory that it may have originated in Persia, and crossed thence into Egypt, Asia Minor, and India. The presence of cotton in the western hemisphere antedates the discovery of America, since Columbus found it growing in a wild state in the West Indies. The natives of Mexico and Peru were found

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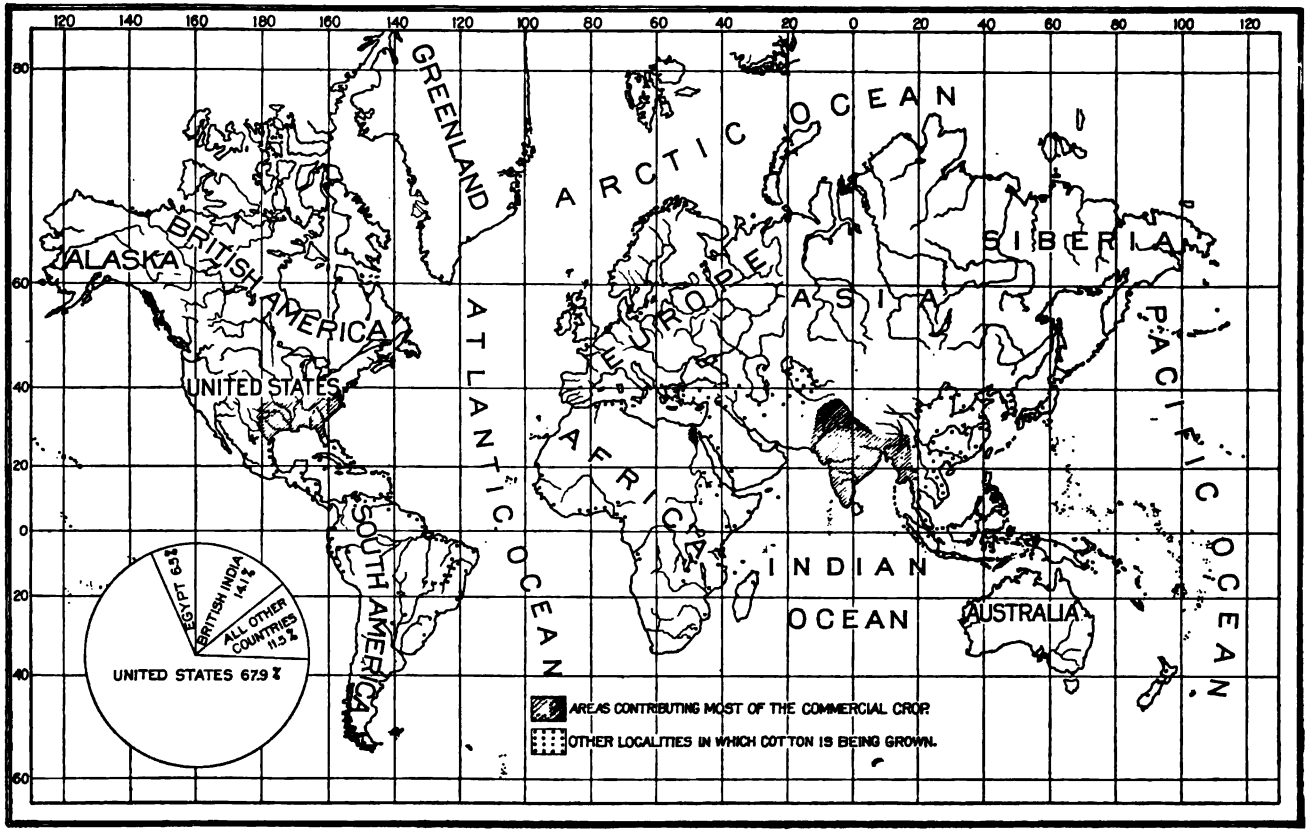

120 140 160 180 160 140 120 100 80 60 40 20 0 20 40 60 80 100 120
80
60
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0
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GREENLAND
ARCTIC OCEAN
ALASKA
BRITISH AMERICA
UNITED STATES
EUROPE
ASIA
SIBERIA
AFRICA
ATLANTIC OCEAN
SOUTH AMERICA
INDIAN OCEAN
AUSTRALIA
PACIFIC OCEAN
EGYPT 6.9%
BRITISH INDIA 14.1%
ALL OTHER COUNTRIES 11.3%
UNITED STATES 67.9%
AREAS CONTRIBUTING MOST OF THE COMMERCIAL CROP.
OTHER LOCALITIES IN WHICH COTTON IS BEING GROWN.

FIG. 1

wearing clothes made of cotton when these countries were conquered by the Spaniards in 1519 and 1522, respectively.

2. Cotton Area and Production.—As the plant thrives only in warm countries, the cotton crop of the world is produced almost entirely between the fortieth parallel, north latitude, and the twentieth, south. Outside of this belt cotton is grown only in localities that are particularly favorable to its cultivation. The area suitable for growing cotton extends from the African shores of the Mediterranean Sea to the Cape of Good Hope; from Spain to Japan and Australia; and from Norfolk, Virginia, in the United States, to Buenos Ayres in South America. Notwithstanding the vastness of the area in which cotton will grow, the regions producing almost the entire commercial crop are the southern part of the United States, India, and Egypt, relatively important in the order named. At the present, time the United States contributes 67.9 per cent. of the commercial cotton; British India, 14.1 per cent.; Egypt, 6.5 per cent.; Brazil, 1 per cent.; and the West Indies, practically none. Of the countries that were prominent in the production of commercial cotton in 1790, Brazil alone retains importance.

3. The localities in which cotton is being grown with more or less success are indicated on the map shown in Fig. 1. The localities shaded are those which contribute most largely to the commercial supply, while dots indicate other cotton-growing localities. So great is the effort to locate new cotton fields that only general accuracy can be claimed for the map. Failure to designate a locality does not necessarily signify that no effort is being made there to grow cotton.

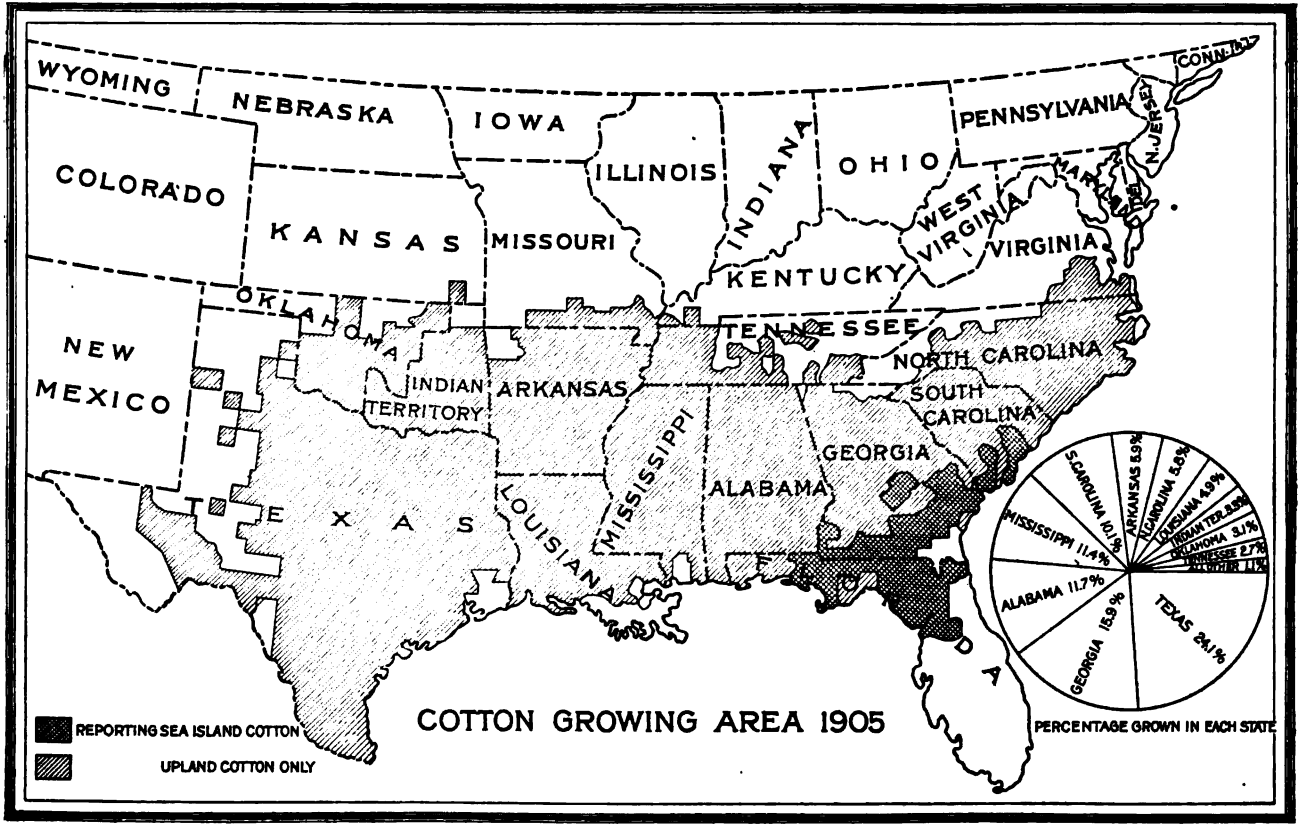

WYOMING
NEBRASKA
IOWA
ILLINOIS
INDIANA
OHIO
PENNSYLVANIA
CONN.
N.JERSEY
COLORADO
KANSAS
MISSOURI
WEST VIRGINIA
VIRGINIA
KENTUCKY
OKLAHOMA
TENNESSEE
NEW MEXICO
INDIAN TERRITORY
ARKANSAS
NORTH CAROLINA
SOUTH CAROLINA
GEORGIA
MISSISSIPPI
ALABAMA
TEXAS
LOUISIANA
FLORIDA
REPORTING SEA ISLAND COTTON
UPLAND COTTON ONLY
COTTON GROWING AREA 1905
PERCENTAGE GROWN IN EACH STATE
S.CAROLINA 10.1%
ARKANSAS 8.9%
N.CAROLINA 8.6%
LOUISIANA 4.9%
INDIAN TERR. 3.5%
OKLAHOMA 3.1%
TENNESSEE 2.7%
MISSISSIPPI 11.4%
ALABAMA 11.7%
GEORGIA 15.9%
TEXAS 24.1%

FIG. 2

DEVELOPMENT AND UTILIZATION OF COTTONSEED PRODUCTS

4. The histories of cotton, cottonseed, and cottonseed oil are entirely separate and distinct. The cotton plant, as well as the use of the cotton fiber for weaving into cloth, has been known for thousands of years, while the utilization of the seed for various economic purposes is of very recent date.

5. The cultivation of cotton in the United States is said to have first been undertaken at Jamestown, Virginia, in the year 1620. At the present time it is grown in all the Southern States, from Virginia to Texas, and may be said to be the greatest industry of the South. The cotton-producing area in this country and also the percentage of cotton grown in each state are indicated on the map shown in Fig. 2.

The seed is generally planted in March and the early part of April. It is thickly sown in rows from 4 to 6 feet apart by machine planters that distribute the seed economically and evenly. The plants come up in about a week, and when they are an inch or two high, they are thinned out so that only one or two stalks remain in a hill. The hills are usually spaced about 1½ feet apart.

Careful cultivation is necessary to the proper development of the plant. The cotton plant is not, as is generally supposed, a hardy plant. It is very sensitive to climatic changes. Too much rain beats off the forming flowers, generally known as *bolls*, and causes too rapid growth, and after the bolls open, it causes "wet-weather blight," or rust. If the season is too dry, the vitality of the plant weakens and the bolls drop off and "dry rust" is caused. The plants develop and grow until killed by the frost. During the period of growth, bolls form and open continuously, thus making the cotton-picking season a long one.

6. After being picked from the open bolls, the cotton is taken to what is known as a *cotton gin*, where, after more or less exposure to dry it, the lint is separated from the

seed by means of saws that revolve at a high rate of speed. The seed is conveyed to the seed house, while the cotton is removed from the saws by means of brushes and carried by a blast of air to the condenser, where it is compressed into bands and pressed into bales for shipment.

While the saw gin is universally used in the United States on woolly seed, in the districts where Sea Island or "black" cottonseed is raised, a form of roller gin is used. In most foreign countries, even where woolly cottonseed is raised, the roller gin is almost universally employed for ginning the seed.

7. There are many varieties of cotton, as large quantities are grown in South America, Egypt, China, and India, and it follows that these countries also produce large quantities of cottonseed. The ginned seed, however, is not utilized in the manufacture of cottonseed products, except in Egypt, where it is thus used to a limited extent. The different cottons produce different kinds of seed, the Egyptian variety being almost identical with the Sea Island seed of the United States, while Indian cotton gives a seed that is about half the size of the ordinary American seed and is covered with a comparatively short lint.

As the cotton plant is grown primarily for the production of cotton, the value of the crop of seed is governed by the demands of the oil mills. In many localities the demand exceeds the supply, and the value of the seed is enhanced far beyond what was thought possible a few years ago.

8. **Development of the Oil Industry.**—The utilization of cottonseed in the manufacture of oil and other economic products has added millions of dollars to the value of the cotton crop, and the steps in the growth of the industry are of interest.

For a long time the Chinese have used ground cottonseed for cattle feed, and incidentally have recovered a little crude oil, which has been used for illuminating and lubricating purposes. The first authentic record of the extraction of oil from cottonseed in the West was in 1783, when seed from

the West Indies was pressed in London with such satisfactory results that a prize was offered by the Society for the Promotion of Commerce and Arts to any planter in the British West Indies who should express a ton of oil from the seed and make 500 pounds of dry, hard cake fit for cattle food. This prize offer was finally withdrawn, as no attempt was made to win it.

In 1832, a small cottonseed-oil mill was operated on a small island off the Georgia coast, and 2 years later attempts were made to extract oil from the seed at Natchez, Mississippi. In 1847, further unsuccessful attempts to extract oil from cottonseed were made at New Orleans; one souvenir bottle of oil is said to have cost the experimenter $12,000. In about 1855, the oil business was revived again at New Orleans, and also at Providence, Rhode Island. Since that date, owing to a study of foreign methods, the business has grown quite steadily. Previous to 1867, there were only four cottonseed-oil mills in the South and only seven in the whole country. The output of these mills was small and the products crude. At the present time there are over 800 mills in the South, each having a daily capacity of from 15 tons upward of cottonseed. Considerably more than one-half of the total cottonseed production is crushed into oil.

9. The manufacture of cottonseed oil on a large scale may be said to date from about 1870. The crude and imperfect methods of that time have given way to modern scientific and business methods. Previous to this time, cottonseed was a positive nuisance, and the disposal of it was a serious problem. It had only a limited use as a fertilizer. Great quantities of it were piled on vacant fields, rendering the soil useless and barren for a time. Sometimes it was dumped into rivers, where it quickly rotted and gave rise to such stenches that laws were enacted to prevent this practice. Much of the seed was disposed of by burning. At the present time the seed is eagerly sought, and it has become a source of considerable revenue to the

cotton grower, and of very great industrial importance to the whole country.

Although the manufacture of cottonseed oil was carried on in England and Holland some time previous to its manufacture on a commercial scale in the United States, this country has outstripped the others both in the quantity and in the quality of goods produced. The advantage that this country enjoys lies in obtaining its raw material in a fresh state, while the foreign millers are mostly obliged to use Egyptian seed, which deteriorates because of the long time required for its transportation to the point of use. English cottonseed oil is made almost exclusively from Egyptian undecorticated seed, from which practically all the lint is removed in ginning. An outline of the English method of manufacturing undecorticated cottonseed cake will be described later.

10. Notwithstanding the fact that great quantities of cotton are grown in India, no cottonseed oil is made there. The Indian cottonseed is of a peculiar nature, being smaller in size than American seed and difficult to delint. Many failures have resulted in attempting to use it. Experiments have been made with delinting machinery, using emery wheels to grind off the lint, but their success has not been demonstrated. There is also a heavy tax in India on manufactured oil, in order to keep the seeds in channels of stock feeding and planting.

Within recent years, however, a practical process has been invented for making decorticated cottonseed cake and choice oil from this small and woolly Indian seed, and at present it is being successfully operated in England. As this process is of commercial interest and may also be applied to any kind of cottonseed, reference will be made to it later.

The shipment of American seed to Europe, especially England and Holland, has met with rather poor success, owing to the bulk of the seed and its great tendency to heat during transportation, which counteract other advantages.

11. Scope of the Cottonseed-Oil Industry.—Formerly the object of the oil miller was to recover only the two principal products, oil and cake, but through scientific chemical investigation it has become possible to recover many valuable by-products from what once was waste.

The tendency now is to concentrate under one management the handling of all products of the seed. It is common for cottonseed-oil refineries to work up their refuse, or *foots*, into various grades of soap, to recover stearin, olein, etc., and to manufacture cooking compounds and other edible products.

The cottonseed-oil business has been well advertised, and few have been more systematically investigated and fostered by the general government. The number of mills is rapidly increasing, but there seems no likelihood of overproduction, as the demand for their products is on the increase, and the prejudice against the use of cottonseed oil as an edible compound has been largely overcome, mainly through the high grade of cottonseed oils produced, the credit for which belongs wholly to the chemist.

12. Application of Chemistry in the Cottonseed-Oil Industry.—To understand the manufacture of cottonseed oil and its various products, a thorough knowledge of all the different materials used in the industry must be obtained and applied. A knowledge of the effects produced by different materials on the various grades of products, as, for example, the effect of caustic soda on crude oil, or the choice of a fullers' earth used to produce white oil, is most important.

Although the successful and economical operation of an oil mill depends to a large extent on the ability of the superintendent to produce a full yield of merchantable product without the aid of chemistry, his efforts will be more reliable if coupled with scientific methods and refinement of working.

13. The field of the chemist in the cottonseed-oil industry is broad and inviting. New possibilities for development are constantly coming up. In the mill, the application of

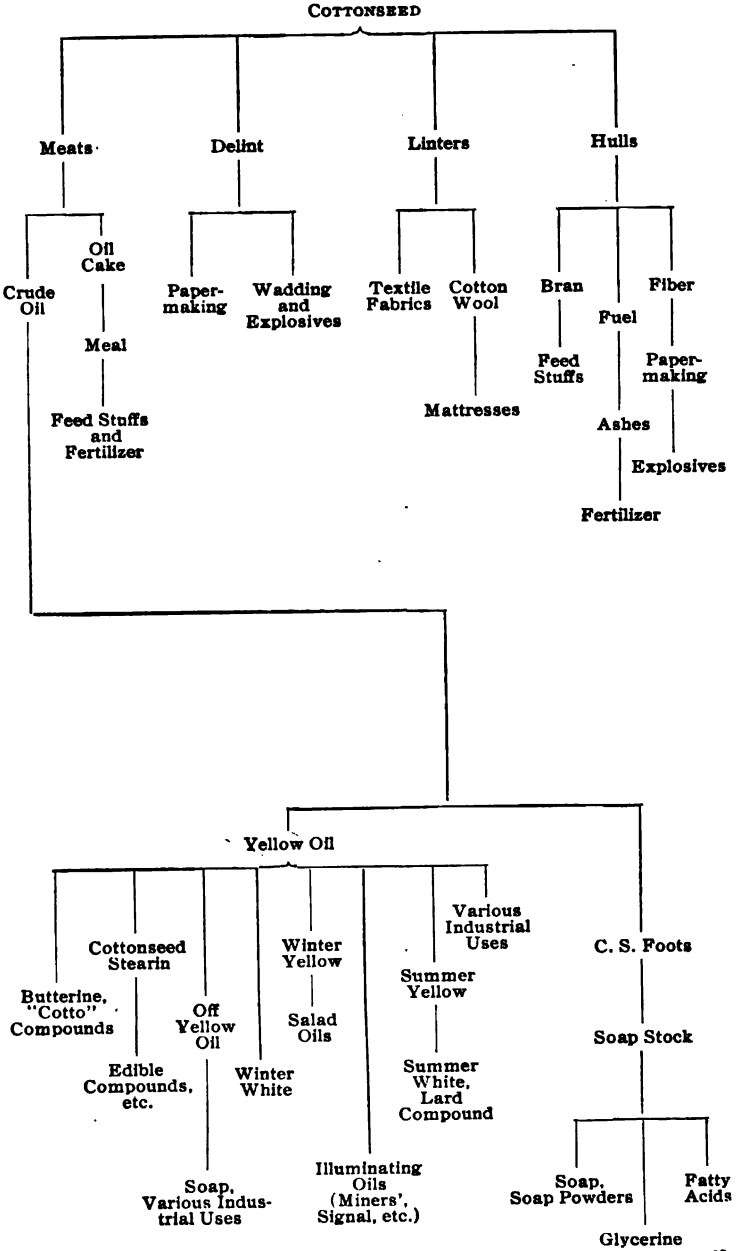

COTTONSEED
Meats
Delint
Linters
Hulls
Crude Oil
Oil Cake
Meal
Feed Stuffs and Fertilizer
Paper-making
Wadding and Explosives
Textile Fabrics
Cotton Wool
Mattresses
Bran
Feed Stuffs
Fuel
Ashes
Fertilizer
Fiber
Paper-making
Explosives
Yellow Oil
Butterine, "Cotto" Compounds
Cottonseed Stearin
Edible Compounds, etc.
Off Yellow Oil
Soap, Various Industrial Uses
Winter Yellow
Salad Oils
Winter White
Illuminating Oils (Miners', Signal, etc.)
Various Industrial Uses
Summer Yellow
Summer White, Lard Compound
C. S. Foots
Soap Stock
Soap, Soap Powders
Glycerine
Fatty Acids

FIG. 3

chemistry in determining the grades of crude oil and the best uses to which the oil is adapted, and in indicating and correcting existing defects, whereby better yields and better products are obtained, attests its paramount importance.

The most important requisite in the profitable production of cottonseed oil and its resultant cake is a modern up-to-date equipment, under intelligent and scientific direction. From the time of receiving the seed, through the various operations of cleaning, decorticating, crushing, cooking, and pressing, careful and systematic working is necessary to produce high-grade, merchantable products. These operations will be described in detail, as will also the further treatment and manipulation by which the primary products are made into various higher grade articles, such as yellow cottonseed oil, white cottonseed oil, and articles of food into which cottonseed oil enters so largely, as butterine, lard compounds, etc. The most recent and approved method for the analysis of the products will also be given.

.The chart, Fig. 3, shows to what extent the cottonseed, as it comes from the gin, is capable of being utilized. The industrial uses are but broadly indicated, further details being given in the text.

COTTONSEED-OIL MILL PROCESSES

PRINCIPLES OF MANUFACTURE OF COTTONSEED OIL

GENERAL REMARKS

14. The operations of the modern cottonseed-oil mill, as the machinery is now erected, are almost entirely automatic; the seed, starting at the sand-and-boll reel, is delivered by elevators and conveyers to the various other pieces of apparatus in turn, no manual handling being necessary until the oil and cake are produced.

Mills of various sizes are in operation, from the small mill of a capacity of 5 or 10 tons per day of 24 hours, for plantation or gin use, to the mill with a capacity of 100 tons or more of seed per day. The mills generally in use are those of one press, 15 to 20 tons, commonly called a *20-ton mill;* two presses, 30 to 40 tons; and three presses, 50 to 60 tons. In each case, the capacity or size of the mill is indicated by naming the larger figure, the capacity being rated in tons of cottonseed that the mill handles. As just stated, the most popular size of mill at the present time is from one to three presses, there being more two-press mills built than any other size.

15. As in other expanding and flourishing industries, recent years have witnessed a tendency toward concentration in the cottonseed-oil industry, the erection of large mills capable of handling several hundred tons of seed daily having been undertaken in localities where the required amount of seed was obtainable. While in many manufacturing industries such concentration is of great advantage, the wisdom

of this procedure in the cottonseed-oil industry is open to serious question. There is a size limit to effective working in every industry, and with cottonseed-oil milling, this limit, in the opinion of many practical men, is in a mill handling about 50 tons of seed daily. Where the latter quantity of material is worked, every detail may be effectively looked after—a requisite for profitable working. When hundreds of tons are worked daily in one establishment, the ever-varying condition of the raw material—the cottonseed—presents so many different phases, together with the many details of all the working operations, that the question naturally arises as to whether or not all the essential details can be given the constant attention necessary for careful, profitable, and thorough working.

Another point bearing on the erection of large mills is the source and proximity of supply of a sufficiently large quantity of seed to keep the mill, when once started, in steady operation.

16. The usual number of hands employed in the oil mill is about twelve to fourteen in a one-press mill, from fifteen to twenty in a two-press mill, from twenty to twenty-five in a three-press mill, and twenty-five to thirty in a four-press mill. This proportion will not decrease much in the larger mills, as there is always more work in handling seed and the products in the large mills. The amount of labor required in a mill naturally varies with circumstances—the class of labor, the arrangement of the mill, the capability of the superintendent, and other conditions.

For motive power in the larger mills, with the modern Corliss type of engine, it is customary to allow from 2 to 2½ horsepower to each ton of capacity, and for small mills, with a slide-valve engine, from 2½ to 3 horsepower for each ton of capacity. Additional boiler capacity must be allowed in all cases for cooking meats and other operations for which steam is required.

17. The general principles that govern the manufacture of cottonseed oil may be briefly summarized as follows:

Thorough screening and cleaning of the raw material, the seed, from foreign matters; thorough and good hulling, or decortication, of the seed; after this operation, as complete a separation of the hulls and meats as possible; perfect and thorough crushing of the meats by the roll; correct and careful manipulation of the crushed seeds in the cooking kettles, with regard to temperature, moisture, etc.; and sufficient and adequate pressure to the cooked meal for the extraction of the oil from the prepared material. The correct application of these principles embraces the whole operation of making from the raw cottonseed the crude cottonseed oil and cake.

In a well-equipped modern mill, the cost of working cottonseed into its products—oil, cake, and meal—is about $3 per ton, the cost varying according to circumstances, equipment, and individual management.

PREPARATION OF COTTONSEED

18. Condition of the Seed.—As it comes from the gin, cottonseed is covered with a soft down that adheres closely to the seed hulls. When removed, this down is called *linters*, to distinguish it from the long cotton fibers separated at the gin and constituting the lint or cotton of commerce. The seed is of a size somewhat smaller than an ordinary bean and is incased in a tough, blackish husk, or hull. The amount of moisture contained in the seed received at the mill varies greatly, the soil, season, and climate influencing the amount to a marked degree; and it is because of this variation in the contained moisture that the methods employed in the most important part of the milling operations, the cooking of the meal, are subject to constant changes. Cottonseed from the first cotton picked or from unmatured or frost-bitten plants gives a small yield of oil of comparatively poor quality. The best results in oil milling are obtained from mature seed.

19. Storing the Seed.—As the seed comes to the mill it is always mixed with more or less dirt, cotton bolls, sand,

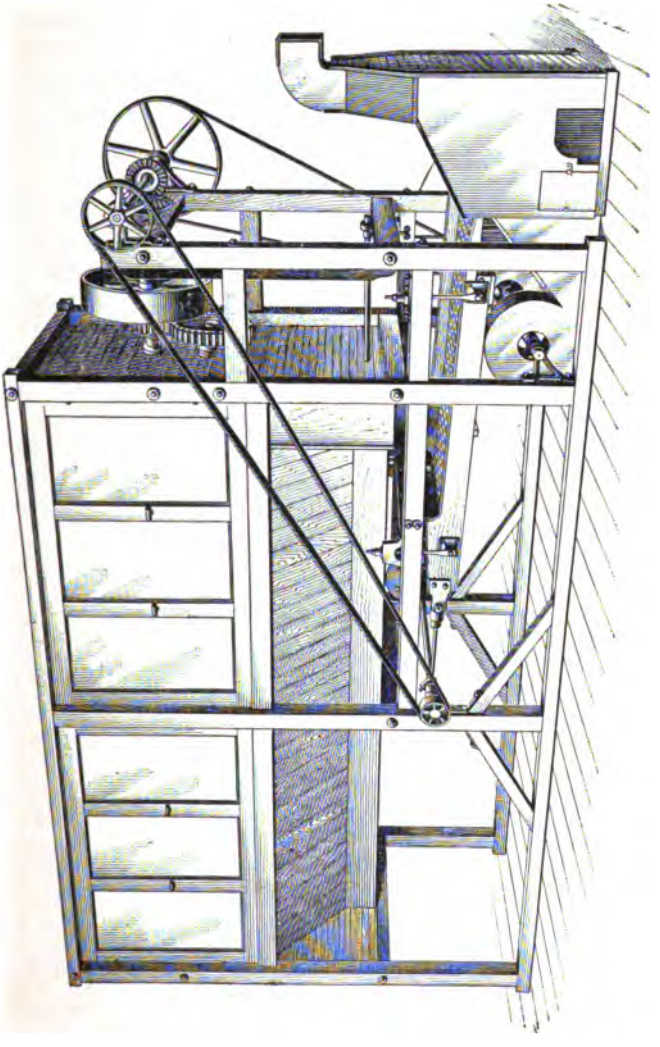


FIG. 4

and other useless material, which must be removed before the seed is subjected to the first process—that of delinting. On being unloaded from the wagons or cars, the seed is carried to the seed storage houses or to the mill direct by means of conveyers having perforated iron bottoms, through which a large part of the accompanying dirt and sand fall to the ground or to another conveyer below that carries the refuse material to some desired place or receptacle.

The loss in weight from useless material in the seed depends very largely on local conditions in cleaning, the loss, if an average may be given, ranging from 3 to 6 per cent. In some instances, this loss has been as high as 10 per cent., which, however, is exceptional.

20. The seed storage houses hold the raw material—the seed—which is secured at the time of harvesting and ginning, for the operations of the milling season, the material being stored in bulk until required for making into the oil and cake. The seed in these storage houses frequently is from 30 to 40 feet deep; but to make compartments holding such a mass is very injudicious, as the great weight pressing on the under seed is very apt to cause the generation of heat and consequent fermentation.

Seed that has been trampled on or partly crushed through handling is very prone to heat from oxidation of the exposed oil, and the danger of deterioration in the bulk of seed with which it is mixed and stored is increased. The more modern plan is to divide the storage houses into different compartments, each holding a moderate amount of seed, thereby lessening the liability of heating and deterioration by reducing the bulk that is kept in one compartment. It is very important to keep the stored seed cool and dry and to have the storage houses provided with good ventilation.

21. Sand-and-Boll Screens.—From the storage room the seed is carried by spiral conveyers, and by bucket elevators where necessary, to the **boll reel**, or, as it is commonly known, the **sand-and-boll screen.** This apparatus is shown in Fig. 4.

It is self-contained and varies in dimensions according to the capacity required. The seed is fed into the reel, in which is a perforated cylinder revolving within a closed box. The openings in part of the steel cylinder are larger than the seeds, which, falling through on a shaker, are evenly distributed over its surface. As they pass over the edge of it, they are caught by a current of air from a fan near the screen and blown over a magnetic or precipitating field, which catches and retains any bits of iron, stone, and dust that remain in the seed. Various forms of magnets are used, including electromagnets, which are probably the most effective. The screen removes from the seed the sand, dust, nails, and other foreign bodies, among the latter being cotton bolls—the capsules of which originally contained the seed cotton. When separated, these substances pass from the screen through spouts into bags or boxes. When the quantity of this material is small, it is picked over by hand and an inferior variety of cotton obtained. This refuse material is technically termed *grabots*, *motes*, or *tailings*. Machines are at present used for reginning this material to obtain from it all the cotton possible.

22. The machine shown in Fig. 4 is a superior form of sand-and-boll screen. This machine is very economical as it effects a great reduction of wear and tear on the linter saws, owing to the almost complete removal of metallic substances. The apparatus is also provided with an aspirator and dust flue for the purpose of removing as much dust and fine dirt as possible. For two-thirds of its length, the reel proper is covered with perforated metal or a screen of proper size to allow the sand and dirt to pass through; the remainder of its length has larger perforations for the seed to drop through. Provision is also made for carrying off the accumulated sand and dirt, usually by a worm, or screw, conveyer. The cleaned seed is carried by the current of air from the fan into conveyer boxes by means of which the seed is automatically conveyed to the linting machines.

23. Grabot Gin.—Fig. 5 shows the **grabot gin,** which has been especially designed for use in cottonseed-oil mills. It is used for ginning the immature bolls, trash, etc. taken from the cottonseed by the screens and cleaners previously mentioned. Technically, this material is termed *tailings.* The grabot gin reclaims from these tailings a merchantable quality of lint cotton, and it is therefore a profitable machine for oil mills to have. It turns into a source of revenue what was formerly thrown away or burned as a complete loss.

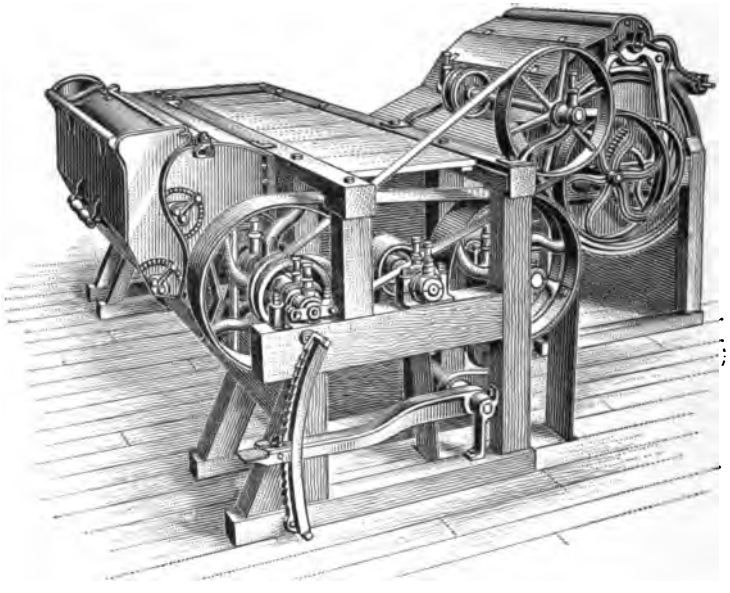


FIG. 5

Gins of this type have fewer saws than the ordinary cotton gin, the usual number of saws in the saw cylinder being seventy. The gin is operated in the same manner as the cottonseed linter shown in Fig. 6, the bat being collected in the usual manner by the revolving drum. In the grabot gin, however, no feeder is provided, as it is much more practicable to feed it by hand. The operation of the linter will be described later.

24. Size of Sand-and-Boll Screens.—Sand-and-boll screens of the different styles vary in size from 3 feet in diameter and 8 feet long to 5 feet in diameter and 18 feet long; when more cleaning capacity is desired than is afforded by these screens, it is customary to use one reel to remove the sand and another to remove the bolls and other foreign matter. The seed is usually first sent through the boll screen, or reel, which separates most of the matter larger than the seed; the latter then goes into the sand screen, in which the sand and dust that remain are quite thoroughly cleaned

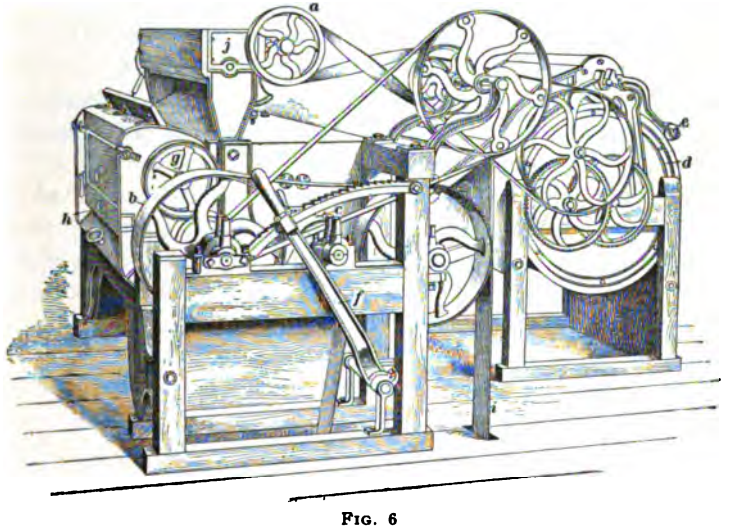


FIG. 6

out. In some mills, the sand-and-boll screens are used in the reverse order, as determined by individual preference, experience, or influencing circumstances.

The wire cloth or perforated steel is adapted in size of mesh to suit the character of seed worked by the mill, as in some sections of the country the seed is small, with but a small proportion of dirt and trash, while in other sections the seed is large and has much sand and dirt mixed with it. The latter is especially true in the Mississippi and Red River bottoms. Where it is customary for the ginners to put all motes and dirt from their condensers into the cottonseed, the

loss in cleaning the seed is sometimes as much as 250 pounds to the ton. Recent regulations, however, have tended greatly to reduce this intentional mixing of worthless material with the seed. The loss in cleaning the seed and working it into its various products will vary from 80 to 200 pounds per ton, depending on individual working systems.

25. Delinting of Cottonseed.—Previous to the year 1887 there were practically only two methods of removing the lint adhering to the hull of the seed, one being a chemical process that destroyed the fiber and the other a mechanical process that crushed the shell of the seed and extracted the kernel. By both of these methods the adhering lint was wasted, being completely destroyed by the former method and rendered useless by the latter because of being mixed with and attached to the broken shell.

The patented chemical process mentioned consisted in carbonizing the fiber on the hull by means of sulphuric acid, permitting its subsequent removal by washing easy of accomplishment. After repeated tests, however, it was abandoned, owing to the expense involved in the operation, the tendency of the sulphuric acid to carbonize and otherwise deteriorate the quality of the meats, and the destruction of the valuable lint.

26. Cottonseed Linter.—The machine used today in most of the mills in the United States is shown in Fig. 6 and is known as the **cottonseed linter.** Through the use of this apparatus, the yield of oil from cottonseed has been very largely increased. At the same time, the quality of the cake has been improved to a marked degree and a merchantable article has been obtained from the short fiber adhering to the ginned seed.

The linter is made with a capacity of from 3 to 20 tons of cottonseed every 24 hours, although claims for a much greater performance are made for some newly patented linting machines. These claims, however, yet remain to be satisfactorily demonstrated in producing a well-delinted seed.

27. The cottonseed linter shown in Fig. 6 removes most of the lint from the seed after it has been through the cotton gin, producing a merchantable quality of cotton known as *linters*. The yield of this material depends wholly on the condition of the seed, wet or dry, the set of the saws, and the amount of seed admitted through the feed hopper. The cleaner the seed is made in the previous operations, the better will be the quality of the lint obtained, and the more work the linter is able to perform without filing and gumming the saws. The modern linter is provided with an automatic feed *a* that may be adjusted to any capacity, to the quality of the lint, and to the condition of the seed. Before the application of this mechanism, an operator was constantly in attendance regulating the feed. When the seed was damp, it would go through the linter very fast; when dry, it would be linted very slowly, thus requiring constant observation by the operator. The automatic feed on a linter, together with a saw filer and gummer, materially increases the daily output of the oil mill.

28. The linter operates as follows: The seed is feed into the linter at *j* and the supply is regulated by an automatic feed actuated by the pulley *a*. The pulley *b* operates the cylinder with the saws (not shown in the illustration). As the seed enters the linter, the supply is regulated for the saws by the mechanism operated by the pulley *g*. As the seed is fed to the saws, the latter, rapidly revolving, remove the lint from the seed. The lint, in turn, is removed from the saws by brushes on a revolving cylinder actuated by the pulley *c* and passes through a flue (not shown in the illustration) to the slowly revolving gauze drum *d*, on which it collects and is wound on the roll *e* to any convenient size. The high speed of the revolving brush cylinder creates a strong current of air, which blows the lint through the flue.

When sufficiently large, the roll *e* containing the linted cotton is lifted bodily from the machine, withdrawn from the center of the cotton, and the roll again replaced on the linter. The seed, after being linted by the saws, falls below

and emerges at *h* ready for hulling. The lever *f* serves to start and stop the linter by tightening or causing to run loose the belt *i*.

29. The apparatus is provided with over a hundred saws arranged on an iron cylinder. From the husk of the seed the saws cut the lint, which then passes through a flue into the condenser. The *bat*, or sheet of loose fiber, here formed is collected into a roll by the swinging roll with which the condenser is provided. The seed passes from the linter with only a slight covering of lint, and is greatly improved for subsequent working purposes, as the huller will do better work on the seed in making a more complete separation of the meats and hulls; better oil and cake will be obtained than from seed not so treated.

Until within the last few years, all attempts to obtain perfectly clean seed in a commercially profitable way were unsuccessful. At present, however, there are machines that remove all the lint remaining after the procedure just mentioned. The short, woolly fiber removed is technically known in the trade as *delint*, or *fiber*. The cleaner the seeds are the more perfect is the separation of the hulls and kernels, or meats; consequently, a better yield of oil is obtained from each ton of seed.

The quantity of lint cotton obtained from the seed, as in other things in this industry, varies considerably; from 20 to 30 pounds are usually obtained from each ton of seed worked. A general average may be taken as 25 pounds.

The amount of seed that can be run through the linter is practically unlimited, but the most profitable results are obtained by delinting from 6 to 8 tons of seed per linter per day of 24 hours.

30. Delint.—By referring to the chart of cottonseed products, Fig. 3, it will be seen that in addition to linters, **delint** also is obtained. This substance, as just mentioned, is the very short, woolly fiber remaining on the cottonseed after the usual linting process. The short fiber is obtained

by practically polishing the seeds, thereby removing all lint except at the small depressed end of the seed. The seed obtained through this process resembles the Egyptian cottonseed, and is known in the trade as *delinted seed.*

31. Delinting Machine.—The machine for delinting cottonseed is a recent invention. It consists of four upright pillars, midway between which is a vertical shaft on which four circular plates are fastened. These plates are provided with a more or less rough abrading surface. Three inches above these plates are circular brushes of the same size, each having a large circular opening in the center. The brushes are held stationary by means of arms attached to the upright pillars, while the shaft to which the plates are attached revolves. A cylinder of sheet zinc incases the shaft plates and brushes, while a hopper between each pair of plates conveys the seed from one plate to the other through the circular openings in the brushes. The seed is fed in at the top of the machine, passes through the aperture in the center of the first stationary brush, and is rubbed between the brush and the first emery plate. In this manner, part of the lint is removed from the seed. This seed then passes outside the circumference of the first plate into another hopper, which sends it through the opening of the second brush to the second emery plate, where the rubbing process is repeated. In this way the operation is performed through the series of the four plates. The lint is removed from the machine by means of a fan, which carries it to the desired place. The cottonseed is thrown out at the bottom of the machine in a highly polished state and entirely free from cotton, except for a minute quantity on the depressed end of the seed.

32. The delinting machine described enhances to a considerable extent the value of the usual delinted seed, as the delint obtained is of considerable value in the manufacture of paper wadding, certain forms of explosives, and even in some kinds of fabrics. With the ordinary delinting process,

this lint is left attached to the hulls, which are always sold at a low price. For exportation purposes, seed of this character, which is known also as *bald* or *black seed*, is far more suitable than ordinary seed, as it occupies much less space and is far less subject to damage from heating and consequent deterioration, owing to the absence of lint.

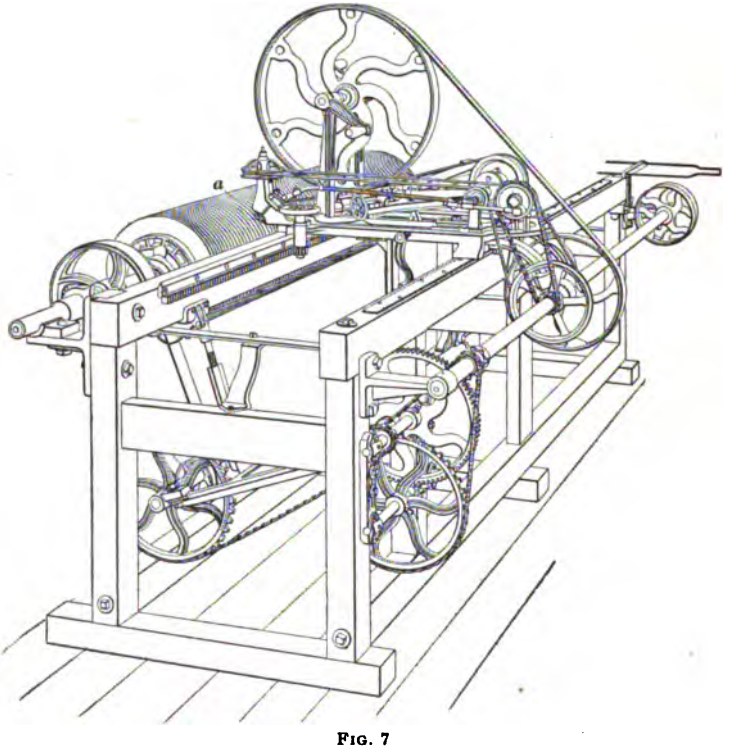


FIG. 7

Another machine for recovering the delint has been invented recently, but up to the present time it has been used only to a limited extent, and only in certain localities. The seed treated by this machine is also almost entirely denuded of lint.

The quantity of delint obtained from a ton of seed has been shown by tests to be about 125 pounds, this being in addition to about 25 pounds of the usual linters from the

ginned seed; but further tests will enable those interested to judge more accurately of the capabilities and advantages of this process.

33. Saw-Filing and Gumming Machines.—In close proximity to the linting machines are the necessary **saw-filing machines** and **gummers.** These machines are in constant use while the mill is in operation for sharpening and gumming the saws of the linters. Like most of the cottonseed machinery, they are practically automatic in action and are a necessary adjunct to every mill in which linters are used. Fig. 7 shows the filer and gummer engaging the saws of a linter, while Fig. 8 shows a view of

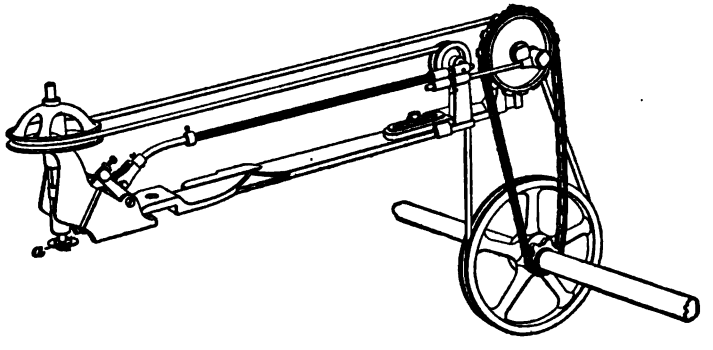


FIG. 8

the detached gummer, or machine for deepening the worn teeth of a saw. The sand and dirt on the seed that escape elimination by the sand-and-boll reel rapidly dull the linter saws, rendering resharpening a frequent operation. In order to perform linting properly, it is necessary that the linters be in perfect condition by having the saws kept sharp.

In working the linting machines, care should be exercised not to overcrowd the machines, as in such cases the lint is very imperfectly removed, with a consequent loss in the yield of linters. Another point depending on this condition is that the more lint that remains on the hulls, the more of the finely crushed meats will be retained by the lint, with the inevitable loss of yield in oil and cake.

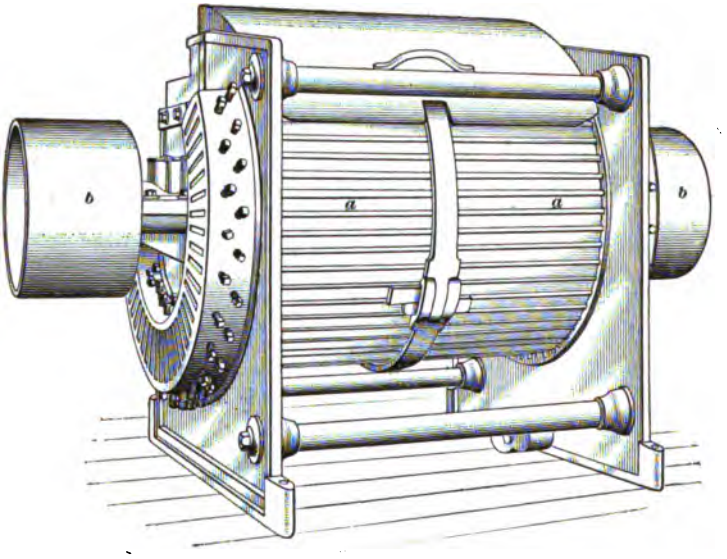


FIG. 9

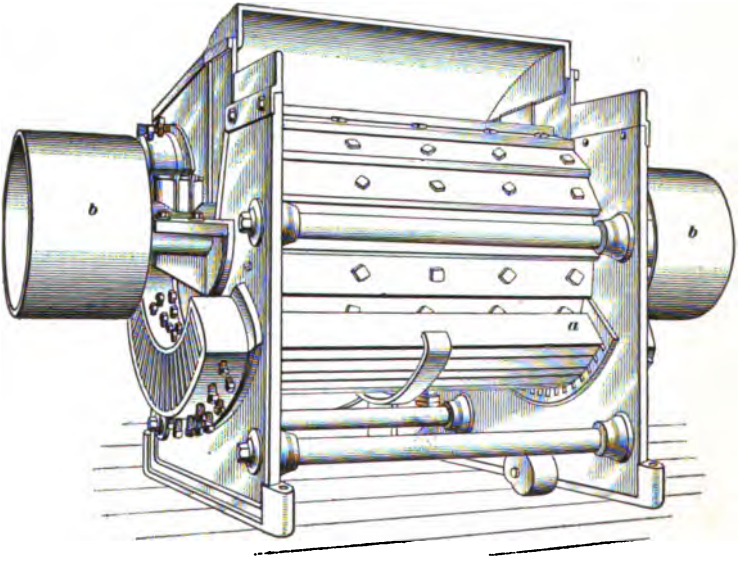


FIG. 10

34. The **gumming** is done by a circular file *a*, Figs. 7 and 8, that rotates on an oscillating spindle and is so arranged that the file can be at work while the saw, which is being gummed, revolves. When the tooth on which the file is working has been sufficiently gummed, the file automatically withdraws and engages the next tooth. The process is continued until all the teeth in the saw are gummed, when the machine stops automatically, and the gummer may be moved to the next saw. Two small, three-cornered files either follow the gummer after the tooth of the revolving saw has been gummed or file the saw before the gummer operates on it. Each method of procedure has its advocates.

HULLING THE SEED AND SEPARATING THE MEATS

35. From the linters the seed is carried by conveyers and elevators to the **cottonseed huller**, a front view of which is shown in Fig. 9 and a rear view in Fig. 10. This machine is conveniently located on the second floor of the mill, and has a capacity varying from 20 to 80 tons of cottonseed every 24 hours.

The hullers consist of cylindrical cases *a* containing horizontal knives that vary in number from eight or nine to thirty, and these knives are fastened on the inner periphery of the cylindrical cases. The cylinders are perfectly balanced, which is a prime essential to the good working of the machine.

The knives in the huller are made of the best steel and are tempered to the proper degree of hardness. Within the horizontal case *a* is a revolving drum on which are fastened knives that, while made to revolve at a high rate of speed by means of the pulleys *b*, barely miss the knives on the inner surface of the cylinder. The seed falling into the drum is rapidly cut to pieces, the mixture of hulls and seed falling to the separating apparatus below the huller.

36. It is of great importance for good work that a regular and uniform feed of seed be maintained at the huller.

Each knife should hull the same amount at each revolution of the cylinder. To meet these conditions, the **cottonseed-huller feeder** is used. This device has an adjustable capacity for feeding any huller, and is so arranged that it can be connected to the hopper just above the huller. By use of a clutch, the supply of seed may be cut off at any desired time, in this way preventing, when the huller is about to be stopped, any choking, thus leaving the huller free for starting up again. This apparatus feeds the seed uniformly and evenly over the surface in the huller.

The knives of the huller are of two kinds. A huller having a capacity of from 75 to 100 tons is provided with seventeen cylinder knives 30 inches long and thirty concave knives. The latter are arranged so that they can be adjusted independently and are held in position by setscrews. The smaller huller has similar knives, but these are fewer in number and about one-half as long. Hullers should be set often and the knives kept sharp, or the seed will not be cleanly cut.

37. Hulling, or more properly speaking, **decorticating,** is, and always has been, a very difficult process, owing to the varying conditions of the seed. When the seed is thoroughly dry and free from excess of lint, the operation of hulling is not so difficult to accomplish, as the hull is then easily broken and the kernel, or meat, is loose and easily drops out of the cracked hull. But when the seed is immature, damp, and soft, the hull will not be broken, but will be mashed so that the meats cannot be separated from the hulls except with a great deal of labor. When the seed is in the latter condition, the knives must be very sharp and the seed fed at a slower rate in order to get a satisfactory hulling.

38. The huller used in decorticating Indian cottonseed does away with the difficulty encountered in hulling wet cottonseed. This huller is constructed without any knives, having instead rods, or arms, that revolve swiftly and act as beaters. After the cottonseed, whether wet, moist, or dry, is introduced into the huller, it passes through two cracking

rolls and then falls into a chamber, where it meets the rapidly revolving beaters and is broken to pieces. Seed that is not crushed is prevented from falling into the beating chamber by means of a narrow grid, through which the beater arms pass in revolving. The seed from the beating chamber falls on a shaking screen that sieves the broken kernels into a conveyer below, and at the same time discharges the hulls and cracked or partly crushed seed into another conveyer. The latter conveyer transfers the material to another huller of the same type, where it undergoes the same treatment.

The huller just described may be worked in a series of two, three, or four, as requirements demand. Damp or wet seed must pass through the series of four hullers, while ordinary, or dry, seed will be decorticated by passing successively through the first and second hullers. The cracking rolls in this type of huller may be adjusted at will. Those on the first huller are usually set far enough apart to admit the seed freely, while those on the other hullers of the series, owing to the fact that a large part of the material is eliminated by the shaker screens, are set closer together.

It has been found by actual working on a very large commercial scale that this type of huller is one of the most effective for decorticating all kinds of cottonseed irrespective of condition, size, or variety. It is automatic in its working, self-contained, and very economical, as it entirely eliminates the expensive knives of the ordinary huller and the labor of frequent sharpening, attendance, etc.

39. In a plant located in Texas, the whole kernels are removed from the cottonseed by a process involving a combination of machinery and chemicals. The capacity of this plant is claimed to be 100 tons of cottonseed daily. The hull-free kernels are mainly exported at the present time. From fresh seed, the clean kernels yield not only a high grade of oil but a much greater proportion of oil than can be obtained from seed decorticated in the usual way; also, as the residual cake is free from the usual proportion of hulls, it contains an unusually large amount of proteid matter.

It has been found by experience, however, that while the hull-free kernels might be advantageously employed for working on the spot, the expense of obtaining them by this process is too great to permit of competition with the ordinary crushing process. Further than this, after a period of transportation the broken kernels are found to be greatly deteriorated and will make only a poor crude oil, as the free fatty acids are unusually high, thereby causing a large and unprofitable shrinkage in refining.

40. Shaking Separator.—When delivered to the **shaking separator,** the decorticated seed from the huller has a considerable amount of the whole meats, or kernels, extracted. This material is sent to the crushing rolls on the floor below by means of a chute. Using a shaker at the huller has been found to be a decided improvement over the practice of sending all the hulls and meats to the meat reel and separator for the entire separation there. By using the shaker, a large proportion of the quantity of meats is removed before the material goes to the separator. Much cleaner meats may thus be obtained, for unless the hulls are so separated, they will be more or less rolled together and intermixed in the revolving separator. In this way, the finely crushed particles of kernels will attach themselves to the lint on the hulls, and thus materially reduce the yield of oil from the cottonseed material.

In some oil mills, there are several hullers, and at each huller is a shaker about 8 feet long and $2\frac{1}{2}$ feet wide. By using this device it is necessary to elevate to the separators only about half the material. Many mills formerly used no shaker at the huller, but elevated the hulls and seed directly to the revolving separators by means of conveyers; but, as previously mentioned, the use of the shaker at the huller is a great advantage in handling the decorticated material. The hulls with the remaining meats in them are then elevated to the meat reel and separator, which is also provided with a shaking separator, and here the remaining meats are extracted from the hulls.

41. Revolving Meat Separator.—The **revolving meat separator** shown in Fig. 11 varies in size to suit the capacity of the mill, the largest mills using machines that are 5 feet in diameter and 18 feet long. The revolving screen is provided with such perforations as are suitable for the class of seed being worked, a mesh of $\frac{1}{4}$ inch being common. Either wire screening or perforated metal is used for the revolving screens. Perforations of $\frac{3}{16}$ inch are used in

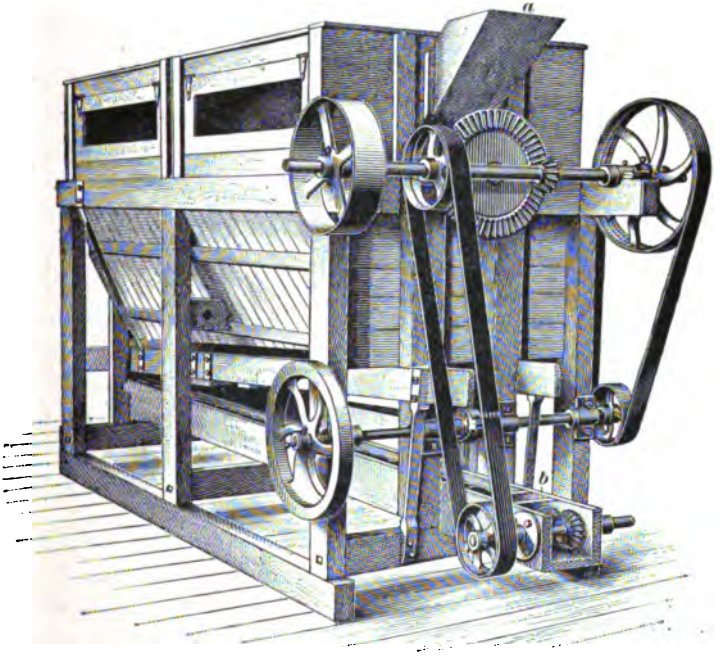


FIG. 11

many screens, but as a rule better results are obtained with $\frac{1}{4}$-inch perforations. Very good work is done by having a screen with $\frac{3}{16}$-inch perforations in one end and $\frac{1}{4}$-inch perforations in the other. The meats and hulls are fed into the revolving reel at *a*, the former falling through the meshes into a spiral-conveyer box *b* below the apparatus. The hulls, owing to their tendency to felt, or to stick together, are retained by the screen and are then delivered to another

separator, the finishing separator or shaker, where more of the meats are sifted out. These screens are arranged in a series, according to the capacity of the mill, the hulls being delivered from one to the other until they are free from meats.

42. Disposal of the Hulls.—When freed as completely as possible from the kernels, the hulls are conveyed either to the hull house, where they are pressed into bales for convenient handling, or to the boiler room for use as fuel. The use of hulls for different purposes will be discussed later. In recent years, the practice of working more hulls into cottonseed meal, to increase the weight of the latter, has prevailed. This manipulation has caused the enactment of laws in several states requiring cottonseed meal, when sold as a feed, to be sold under a guaranteed analysis, following the lines laid down for the sale of commercial fertilizers.

43. As just stated, the ground hulls are frequently mixed with the cakes made from decorticated seed. This practice is followed extensively at the present time, mostly, however, with products intended for domestic use, the export trade demanding a protein content in cake or meal of at least 44 per cent., which amount cannot usually be obtained when ground hulls are added to the cake.

The average protein basis of sales for domestic cake or meal is 41 per cent., this lower percentage being due to the intermixed ground hulls. Ground hulls, or hull bran, is now a regular article of trade and is of recognized value as a feed stuff. It is sometimes mixed in varying proportions with meal from decorticated seed and sold as a feed at a few dollars per ton less than the meal itself. At times, this mixture is pressed into the form of a cake and exported under specific brands.

44. While in former years the hulls were considered of no value and were disposed of in any convenient way, they now have a steady market value as a feed stuff, not only being recognized as an excellent cattle food, but being used

extensively for that purpose. The hulls are either sold loose at the mill or made up into bales weighing about 100 pounds,

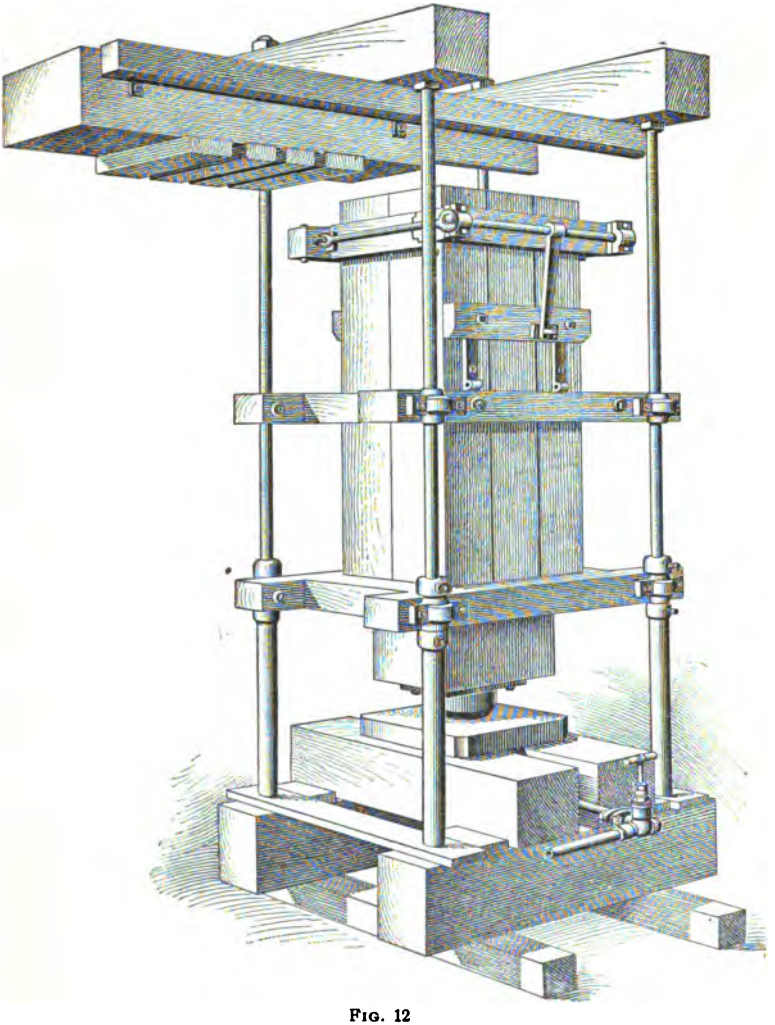


FIG. 12

which is a convenient size for transportation. The loose hulls are compressed into bales by means of a *hull packer*, Fig. 12, which is similar in shape to a cotton-baling press

and operates in about the same way. The packer shown here is run by hydraulic power. Hull packers are also made with a division through the center of the box and with two top followers, so that two bales instead of one can be made at each pressing.

CRUSHING THE MEATS

45. Crushing Rolls.—After the cottonseed meats are separated, they are conveyed to **crushing rolls** such as are

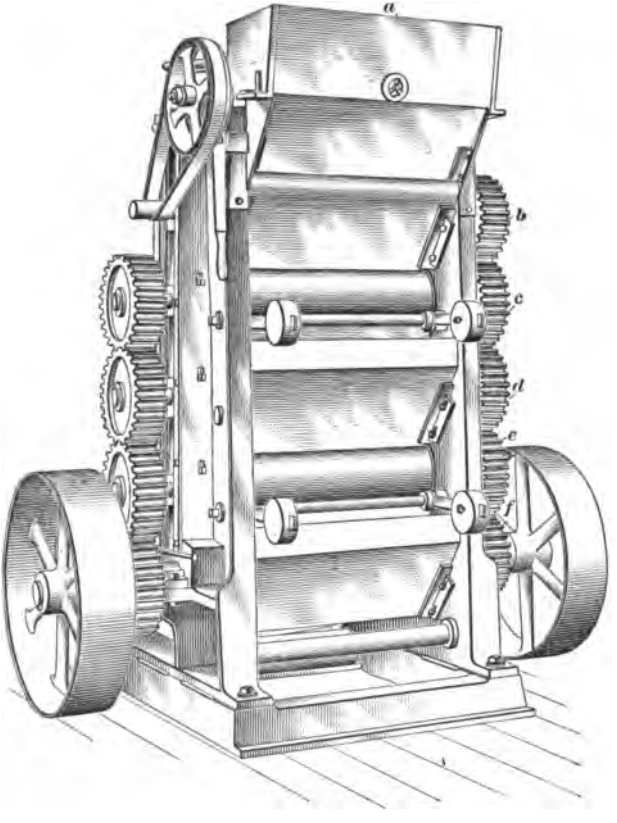


FIG. 13

illustrated in Fig. 13, which shows a set of five high-geared rolls. The rolls themselves, the gears of two of which are

shown at *c* and *e*, are of chilled iron and vary in length, a common and usual length for a 75-ton mill being 48 inches, and for a 100-ton mill, 60 inches. The bottom roll of the set is 16 inches in diameter and the upper ones 14 inches. This arrangement of heavy rolls at the bottom is used in order to assist in driving the upper rolls by friction, which materially lessens the work on the belts and gearing. Each roll is driven by gearing or by belts from both sides; in the latter case the belts are provided with a belt tightener at each side. In a mill of moderate size the machines have from three to four rolls. The roll shafts, with the exception of those of the bottom roll, rest in accurately fitted boxes, so that they are firmly held in position by the heavy iron frame containing them.

Three of the five rolls, the gear-wheels of which are shown at *b*, *d*, and *f*, revolve in one direction, while the other two, *c* and *e*, which are between the first and third and the third and fifth, revolve in the opposite direction. The seed delivered into the hopper *a* at the top passes a small fluted, or corrugated, roll, which distributes the meats uniformly over the entire length of the upper roll, and by means of the guide plate is directed between the first and second rolls. The revolving cylinders, with the inward motion, rapidly draw in and crush the material, causing it to pass between the rolls to the opposite side. The crushed seed is again deflected by the slanting position of the cant board and made to pass between the second and third rolls, thus crushing it again in its passage. This operation is helped by the weight of the two upper rolls. In a similar manner, the seed is carried through the lower rolls.

46. When passing between the lower rolls, the seed receives the aggregate weight of the four upper rolls, which crushes it to a very thin, flaky fineness, thoroughly breaking the oil cells in the meats, a condition essential for obtaining a full yield of oil when the cooked meat is subjected to hydraulic pressure in a subsequent operation. The rolls are provided with balanced steel scraper knives that prevent any

crushed meats from adhering to the rolls. The rolls are sometimes held together by heavy coil springs that assist in crushing the oil cells of the meats and yet allow a hard, foreign substance, accidently present, to pass through without injury to the rolls. The rolls revolve at various speeds, depending on the individual judgment of the operator. From 120 to 180 revolutions per minute is the ordinary range, but as a rule, the best work is done with the latter speed. In a few mills the different rolls are run at different speeds, it being claimed that a better crushing effect is obtained by this method; however, the usual way is to have all the rolls revolve at the same speed.

47. It is very essential that all the rolls be true and in perfect alinement, or good work cannot be done. It is also equally important that all hulls be perfectly eliminated; in crushing any of the latter with the kernels of the seed, the meats will be imperfectly disintegrated, many of the oil cells remaining unbroken, and it will not be possible to obtain the full yield of oil from the seed. The mealy, crushed material should come from the roll in very thin flakes, with the oil cells of the seed thoroughly broken and mashed. The condition of this material should be frequently determined, and a close watch should be kept on the working condition of the rolls. By examining the crushed seed with a magnifying glass, the completeness of the crushing may be easily observed. It is the crushing operation that forms the basis for a good or a poor yield of cottonseed oil, because if poor work is done at this point no subsequent operation can remedy it. The heavier the rolls and, within limits, the more there are, the better will be the crushing; also, when the meats are cooked and pressed, the better will be the yield of oil.

COOKING THE MEATS

48. The next operation, that of **cooking**, which brings the meats into such a condition that, on pressing, the oil is readily liberated, is the most difficult and at the same time

the most important of all the operations of cottonseed milling. There is no universal, or even usual, method of performing this operation, as each individual has his own way of handling the cooking kettles, or heaters, in order to produce, in his judgment, the best results. Such exercise of judgment is necessary in this industry, owing to the fact that the physical characteristics of the seed are constantly changing. As the crushed meats, or meal, as it is often called, is fed to the kettles, one period may be passed with meal in a comparatively dry condition, when suddenly it will change to a moist condition. This may be the case when a fresh lot of seed has been crushed, or when seed from a different section of the country follows the first lot; also, when a lot of seed that has been exposed to rains is crushed, difficult cooking is the result. Should the same treatment be given any of these qualities of meats as was accorded the comparatively dry material, there would be an inevitable loss in the earnings of the mills. The success attendant on cottonseed-oil milling depends in a great measure on the care that is exercised during the cooking.

The treatment of the crushed seed in the cooking kettle, or heater, is a matter of the greatest importance, and the strictest attention is always necessary, as this is the most critical point on which the profits of the mill depend. Carelessness or neglect in cooking nullifies, to a great extent, the operations of rolling, etc., however efficiently these may have been performed. Proper temperatures must be maintained in cooking, and there must be present the correct amount of moisture necessary for the crushed meats to yield the full quantity of oil. As before mentioned, these conditions must be governed by the nature of the seed under treatment, the heat and moisture being varied to suit existing conditions.

The method of cooking the average seed will be described, together with the construction of the cooking heater *a*, Fig. 14, and the subheater *b*. The method about to be described, which has been adopted by many successful mills, has been found to produce excellent results, a yield

of 43 gallons of oil per ton of seed frequently being obtained. The crushed meats from the rolls are carried by conveyers and elevators to hoppers connected with the heaters, the hoppers being so arranged as to hold just a proper charge of meal for the presses.

49. Cooking Heater.—The **cooking heater,** shown at *a*, Fig. 14, is made in one solid casting and is steam-jacketed on the sides and bottom. The sides are covered in

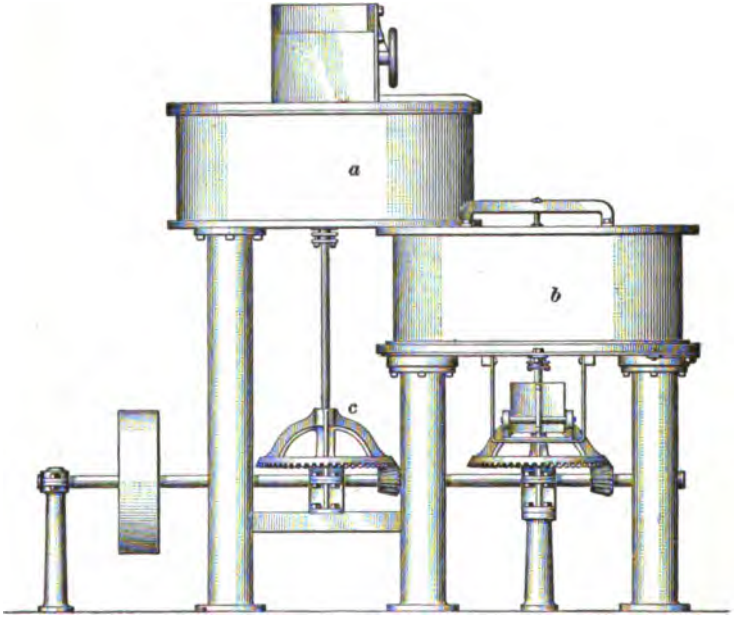


FIG. 14

many instances with insulating material, such as asbestos-faced hair felt, held in place by an outside lagging of sheet iron. The heater is mounted on heavy iron frames, or legs, and is connected with a gear *c* to drive the stirring apparatus, mentioned later, from underneath. Cooking heaters are also provided with an overhead gear for driving the stirring apparatus, the arrangement being a matter of individual preference. Each heater is also provided with a charging hopper that has a hinged and sliding gate for filling the apparatus with

material for cooking. The top of the heater is made in two sections, one of which is hinged and can be raised or slid to one side at will by the heaterman, or cooker, to enable him to satisfy himself as to the condition of the charge and also to give him access to the interior of the heater. The heater is also provided with a stirring apparatus connected with the gear *c*, as just mentioned, and consisting of a heavy, forged-iron stirrer with arms that revolve for the purpose of keeping

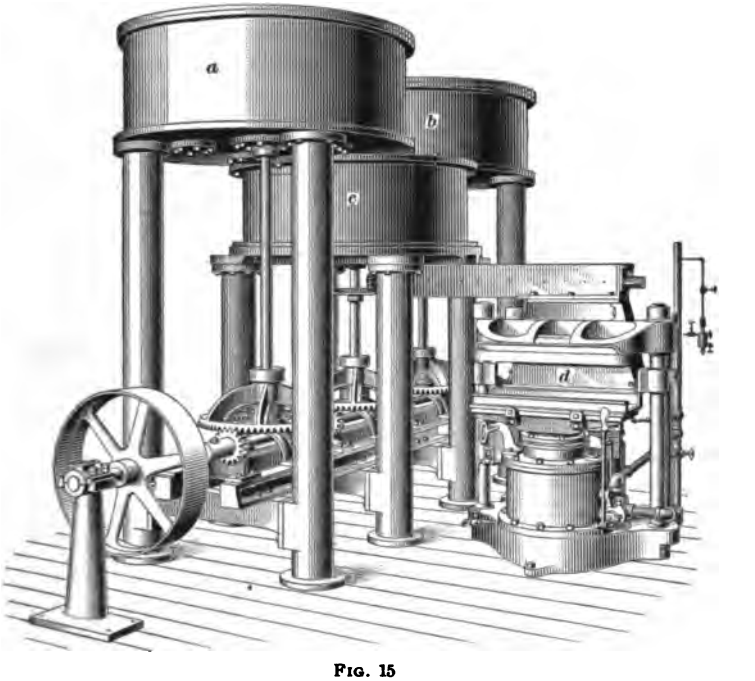


FIG. 15

the meal agitated and preventing it from sticking to the sides or bottom of the heater. This agitation is most essential, for without it the meal would be burned in one part and undercooked in another. The heaters are also furnished with mechanical appliances for breaking up the meal, which forms in balls, technically known as *water balls*. One style of device for this purpose consists of a set of knives that hang into the heater from the top; the balled meal, meeting these

obstructions, is broken into pieces, so that an even cooking may be obtained. In the heaters is a perforated steam pipe for introducing moisture into dry meal when this is necessary.

Cooking heaters are made in two sizes, 52 inches in diameter and 72 inches in diameter. The former has a capacity of 20 tons of cottonseed per 24 hours, and the latter a capacity of 40 tons of seed per 24 hours. The heaters are usually about 18 inches deep.

50. In Fig. 15 is shown the usual arrangement where two heaters *a* and *b* and a subheater *c* are employed. The advantage of this arrangement is that while the meats are cooking in *a*, the finished product from *b* is being emptied into *c*, from which, in turn, the cooked meats are transferred to the cake former at *d*. It will be readily seen that in this way cooking is always going on in one heater while the other is being emptied and refilled; thus, a large number of oil presses are constantly kept busy.

51. Operation of Cooking.—The method of cooking in the heater, after the meat is delivered from the hoppers, depends entirely on the judgment of the heaterman, or cooker. A charge of about 700 pounds of crushed meats, or a quantity adapted to the size of the presses, is placed in the heater by pulling the lever of the hopper, covering the bottom to a depth of about 14 to 16 inches. It is not advisable to have the layer of material too deep, as it is much more difficult to cook evenly. The speed with which the stirring arms revolve in the heater depends on their number and on the amount of material in the heater. A speed of from 45 to 60 revolutions per minute is a good average for a heater with three or four arms, although some are operated at a speed of 35 to 40 revolutions. If the motion of the stirrers is too slow, the meal will get too soft and mush-like and will absorb too much moisture when steam is being injected. The stirrers revolve close to the bottom of the heater, thoroughly mixing and stirring the meal as they revolve. The meal is constantly watched by the

operator, who tests it from time to time to ascertain its condition and admits, by means of the perforated steam pipe, a sufficient amount of moisture to hold the material together when pressed.

The best results in cooking the crushed meats are obtained by using steam at a pressure of about 80 pounds. The time required varies with different classes and conditions of seed. For comparatively dry seed, a cooking of 14 minutes at this pressure gives excellent results. The time necessary for cooking dry meats varies from 12 to 20 minutes, and seed that is damp requires about 30 minutes without any addition of steam to it while cooking. Wet and frosted seed may require from 40 to 50 minutes. Either too little or too much cooking gives a small yield of oil, so that the importance of right cooking is apparent. An undercooked charge contains too much water and, in addition to this, the oil is not made sufficiently limpid and hot to flow freely from the material when placed in the press. If there is an excess of moisture present in the cooked meats when placed in the press cloths, the latter are burst when subjected to pressure, causing much damage to this expensive material. If the material in the heater is overcooked, the oil, although of good yield, will be dark and of inferior character, a rank odor and taste frequently being imparted to it by the overheating. The cake made from overheated or burnt meal is also dark in color and of poor quality. A test frequently applied by the operator is to withdraw a sample in his hand and press it together to ascertain whether it will hold together when tightly squeezed without oozing water. If the meats hold together, they are considered sufficently cooked. If the material will not hold its shape, but falls to pieces, more moisture must be added to assist in forming a firm cake. Knowledge of the proper cooking of meal, like the judging of the quality of cottonseed oil, can be acquired only by practical experience. When properly cooked, the meats are withdrawn either into the subheater *b*, Fig. 14, from which they are fed directly to the cake former, or into a conveyer that carries them to that machine.

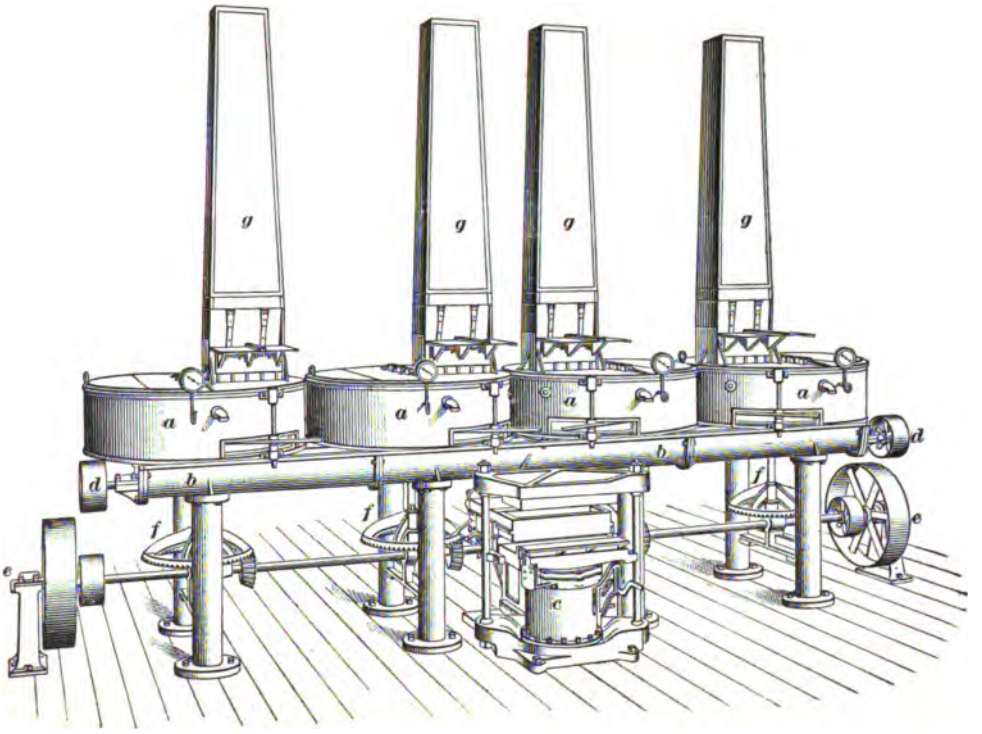


FIG. 16

52. Subheater.—The **subheater** is similar in shape and appearance to the cooking heater, the sides being covered with insulating material, such as asbestos-lined wool felt. It is used for holding the cooked meats preparatory to making into cakes by the cake former. When sufficiently cooked, the material may be dropped directly into the subheater and kept there for a considerable time without injury and at the proper temperature for pressing.

By the use of the subheater the whole charge from the cooking heater may be withdrawn at once, thus obviating all danger of having the last part overcooked when withdrawn directly for use in the cake former. There is, however, a wide divergence of opinion among oil millers regarding the utility of the subheater, some claiming better results without its use; but in this, as in many other operations, individual experience is always consulted. For small mills, the subheater is practically a necessity.

53. Direct-Conveyer System.—Many oil mills use the cooked material without the intermediate holding in the subheater, employing the **direct-conveyer system** shown in Fig. 16. This method eliminates the charging heater, the cooked meal from the heaters *a* being dumped into the iron conveyer box *b* and carried directly to the charging hopper adjacent to the cake former *c*. The conveyer box is provided with conveyer screws, actuated by the pulleys *d*, which carry the meal to the charging hopper and cake former *c* under the conveyer, where it is pressed into cakes.

The pulleys *e*, with shafting, furnish the power to the gears *f*, the latter transmitting motion to stirrers in the cooking heaters. The steel charging hoppers *g* are used to hold the rolled and crushed meats preparatory to being charged into the cookers or heaters. The set of four heaters shown in this figure can be extended to suit any capacity of mill by simply adding other heaters and the necessary length of conveyer.

54. Water Balls.—The formation of **water balls** in the heater and subheater during the cooking is a great

source of trouble and annoyance to the oil miller. When present in any considerable quantity, they cause the cottonseed cake containing them to have a spotted appearance. These balls of meal vary in size from ¼ or ½ inch to 3 or 4 inches in diameter, and are made by the meal forming a nucleus and picking up more meal in rolling, increasing in size, like a rolled snowball, until they can get no larger. The mechanical contrivances in the heater for breaking these balls do not always serve their purpose. These devices, however, are assisted in their work of breaking up the balls by the conveyer in bringing the cooked meal to the cake former and also by the transfer of the material from the heater to the subheater, when the latter is used.

The water balls are practically raw meats on the inside, escaping cooking throughout. They are caused by excessive moisture in the meats. Many devices have been tried to eliminate them from the cooking of the meal, but up to the present time none has been entirely successful. When these uncooked balls are pressed into the cake, the latter has a tendency to become quickly fermented and sour from the decomposition of the uncooked meats.

FORMING AND PRESSING THE CAKE

55. Cake Former.—When sufficiently cooked, the crushed meats are withdrawn from the heater through a door or gate into either the subheater or the conveyer box, from either of which the material is taken to be formed into cakes for pressing. For forming these cakes, the apparatus shown in Fig. 17, known as the **cake former,** is used. This machine, receiving the meal from the cooking heaters, forms it into cakes of the proper size to be inserted in the boxes of the presses. The rams *a* operating these machines are of various sizes, that in Fig. 17 being 18 inches, and are provided with an automatic cushioning device for the drop or return stroke. On the top of the ram is placed a meal box *b* that is filled by a sliding meal carriage *c*. This carriage registers with the discharge hopper of the heaters and is

operated by a small steam cylinder *d* in the rear, making its forward stroke at the will of the operator and its return stroke automatically. By means of the automatic cake former, a 15-box oil press can be filled in less than 2 minutes. The machine works with clocklike regularity, and, standing about waist high, is operated by steam pressure by means of a lever *e*; *f* is a counterbalance.

56. Forming the Cake.—A press cloth of camel's hair or wool about 6 feet long and sufficiently wide to fit the press plates accurately is placed over the tray of the former,

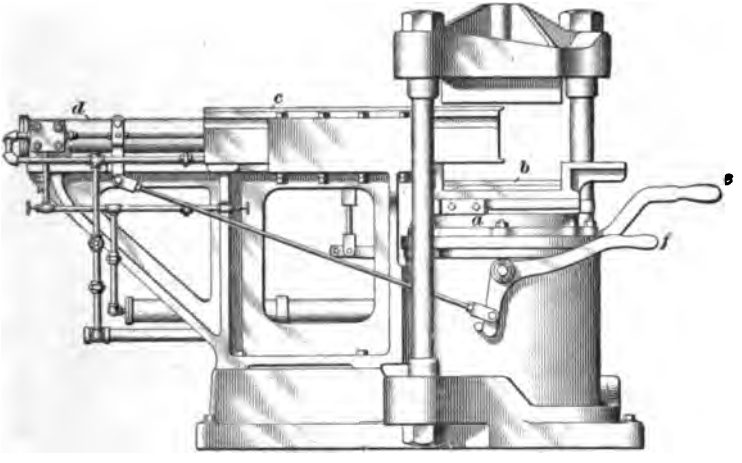


FIG. 17

the center of the cloth being at the center of the tray. The meal is then charged into the tray and cloth, 3 or 4 inches deep, the ends of the press cloth quickly folded over, and the pressure applied, when the cylinder ascends, raising the load and compressing it into a cake. After a few seconds the pressure is released, when the cake pressed in the cloth descends; the cake, with the cloth, is then quickly inserted between the plates of the hydraulic press by means of a thin sheet-steel holder, which is the same width as the tray and somewhat longer than the cake. As fast as made, the cakes are placed, one after the other, after pressing in the cloths,

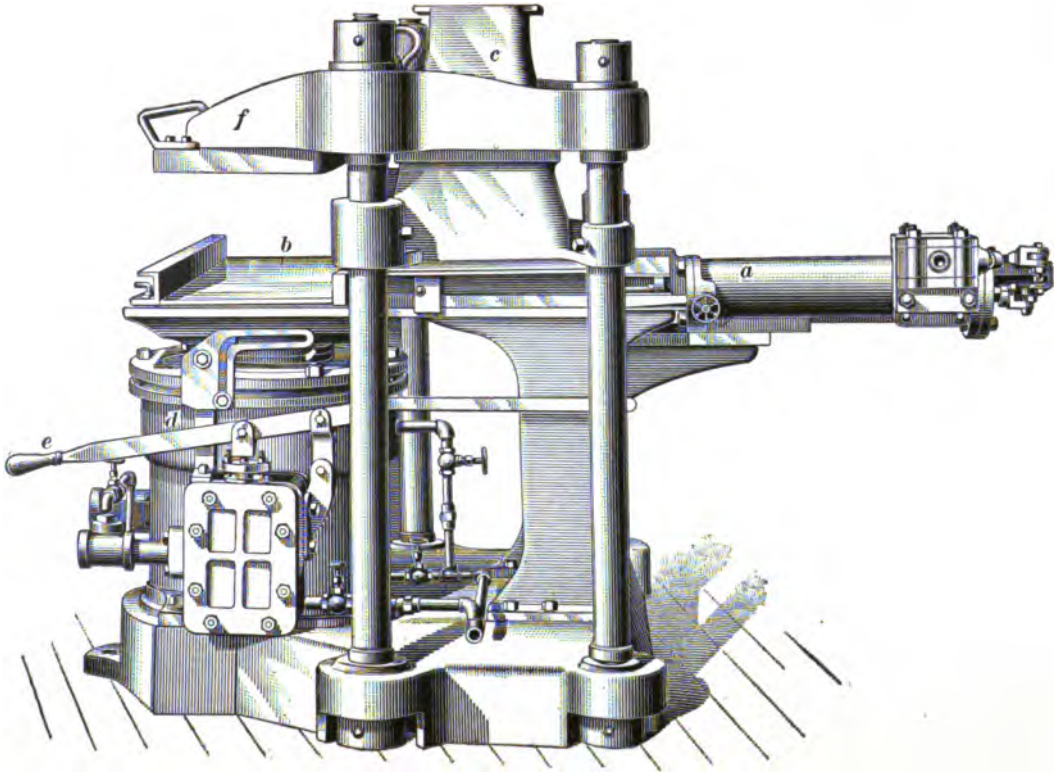


FIG. 18

into the different plates of the press. The whole operation occupies only a fraction of the time required to describe it. The cakes so molded by the automatic cake former are quite perfect in form, thus facilitating their adjustment in the oil press, and being even and uniformly pressed, give a greater yield of oil than was obtained in former years, when the old-style box was used. Hand cake formers are still in use in some small mills, but they are rapidly being superseded by the automatic formers. One machine of this description will furnish a sufficient supply of cakes for from four to six presses as a minimum.

57. Automatic Steam-Puller Former.—The cake former shown in Fig. 18 is among the recent improvements in cottonseed machinery. The automatic former is located under the subheater, and the hopper is situated directly under the gate of the subheater. In operating this machine, the gate of the subheater remains open and the meal comes down into the hopper *c*, filling it. The cloth is placed on the former tray *b* as usual, and a lever (not shown in the figure) operating a valve on the auxiliary steam cylinder *a* is pulled out. The tray *b* moves back under the hopper *c*, fills, and then returns to its position automatically; that is, these operations are effected by the simple act of pulling the lever operating the valve on the steam cylinder. The pan is uniformly filled and struck off. As soon as the tray *b* moves out of its position, it locks the valve of the main cylinder *d*. The return of the tray to its first position automatically unlocks the main cylinder valve; the press cloth is folded over the meal, and the main lever *e* is slightly lifted, as on account of the very long stroke there would be danger of shooting the tray up to the former head *f* under the full steam pressure.

After the tray has made a portion of its upward stroke, the lever is raised as high as possible, giving full steam opening with full pressure for the final forming of the cake. In other words, by partly lifting the lever *e*, the main cylinder *d* with the tray slowly ascends to the former head *f*; when this

position has been reached, the lever *e* is raised to its limit, thereby causing the tray to be pressed against *f* with the full force of the steam, thus squeezing the cooked meal wrapped in the cloth into a compact cake.

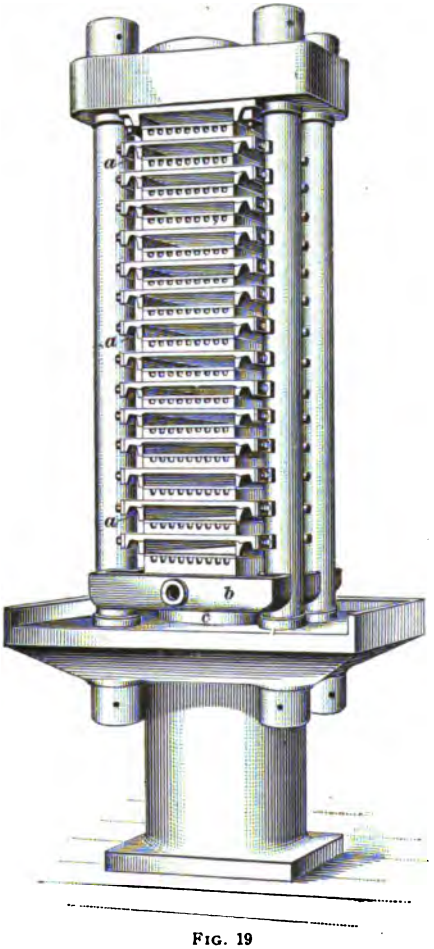


FIG. 19

By pushing the lever *e* down, the steam pressure is gradually shut off and the cylinder with the tray slowly descends to its original position, as shown in the illustration. As the tray descends, a steel sheet is inserted under the cake, and the latter is lifted out and placed in the hydraulic press.

After the tray has reached its original position, another cloth is spread on the tray, the lever on the auxiliary steam cylinder *a* is pulled, and the tray travels back to the hopper *c* to be filled again.

58. Oil Press.— The **oil press** is also a very important piece of apparatus to the oil miller, the pressing being the stage where all previous operations show their efficiency or deficiency. Several styles of hydraulic presses are made, one of the most modern, a 16-inch steel press, being shown in Fig. 19. Hydraulic oil presses are usually made in two sizes, the 16-inch (meaning the size of the ram or piston *c*,

Fig. 19) and the 12-inch. The former size contains from ten to fifteen steel plates *a* and the latter from seven to twelve plates, similar in design to those of the 16-inch, but smaller in proportion. Two views of a steel press plate used in these presses are shown in Fig. 20.

The press, Fig. 19, is made of heavy castings to withstand the pressure required, and is provided with raised edges *b* at the bottom for catching and retaining any oil that may drop down the sides while pressing. For observing the applied pressure, each press is provided with a hydraulic gauge, also a gauge pipe and a valve for its operation.

The press plates shown in Fig. 20 have on the top (*a*) arrangements for the drainage of the oil. The lower side of a plate is shown in (*b*). In this plate there are only two

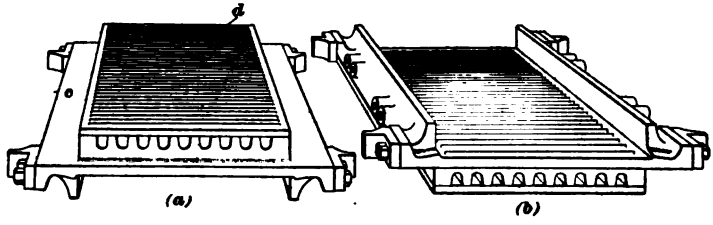


FIG. 20

parts, the main body *c*, made of one piece, and the "drainage plate" *d*, which is attached to the former piece by screws. The drainage may be of several kinds on the press plates, there being a great diversity of opinion as to the best form. The corrugated steel pan $\frac{1}{4}$ inch thick, perforated with $\frac{1}{16}$-inch countersunk holes, is probably as good as any. Some oil millers use a drainage plate with slots sawed out of a solid piece of steel or brass. In any event, the drainage plate must be kept perfectly free of loose meal or other matter, in order to give free passage to the oil from the pressed meal.

59. The capacity of a press depends on the weight and number of cakes. The usual time that a press is run is 20 minutes, or 72 charges in 24 hours, but more than this is often accomplished. When the meal is well and properly

cooked, the cakes average from 12 to 14 pounds each, equal to about 20 tons of seed per day. It is also possible to run the presses on 15 minutes' time, or to make the cakes heavier. The latter course is not advisable, as better results are accomplished with a cake weighing about 14 pounds. The capacity of the hydraulic oil press depends also to a large extent on the capability of the operators and the percentage of oil designed to be left in the cake, as well as on the loss sustained in the amount of press cloth damaged by excessive pressure applied.

60. Pressing the Cake.—The press being filled with the pressed cakes, the operator turns on the hydraulic pressure, and the ram rises and presses the material in the cloths with a pressure varying with different operators from 2,000 to 4,000 pounds per square inch. The lower cakes in the boxes are raised against the upper, and these against the heavy top plate of the press. As the mass becomes more and more compact, the oil begins to flow, at first slowly, and as the mass receives more pressure, the oil gushes forth from the material in streams. It passes through pipes made to receive it into large reservoirs beneath the press, from which receptacles the oil is pumped into settling tanks and, in the modern mills, through the filter press, which will be described later.

After the full pressure has been reached and most of the oil has been pressed out, a period of from 7 to 10 minutes is allowed for the press to drain, when the pressure is withdrawn by reversing the control valve. The ram descends, carrying the lower parts of the press with it. The pressed cakes of meal, still hot from the cooking, are now withdrawn from the press and stripped of the press cloth. After further treatment by drying, they are ready to be packed as cakes or ground into meal.

61. The crude oil, where no filter presses are used for clearing it, is pumped to storage tanks and allowed to settle for some days. Ordinarily, from 3 to 6 days is required to effect a good settlement of the oil, but this, like many other

operations in the oil-milling industry, is a matter largely governed by circumstances. The further treatment of the crude cottonseed oil for industrial purposes will be described later.

62. Hydraulic Pump.—As already stated, the pressure is applied to the oil press by **hydraulic pumps** made especially for this purpose. The pump is always provided with an automatic regulator and safety valve. The hydraulic-pressure regulator serves to admit only enough steam to move the pump at an even speed; as the hydraulic pressure

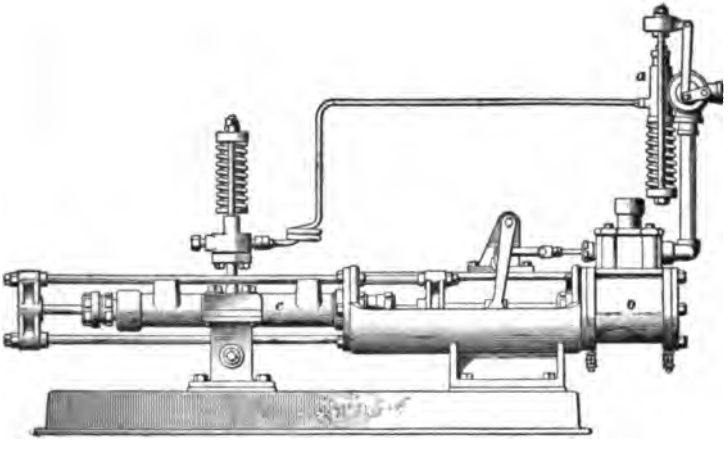


FIG. 21

rises, more steam is automatically admitted to the pump until the required pressure is reached, when the pressure regulator automatically closes and the pump stops, the steam being entirely cut off. The pump thus requires very little attention. Fig. 21 illustrates a modern hydraulic pump; the automatic pressure-regulating valve *a* is shown at the right, and one of the steam cylinders is shown at *b* and a water cylinder at *c*.

63. Accumulator.—Most cottonseed mills are equipped with an **accumulator,** which is a device for maintaining the pressure generated by the pump. The accumulator is used

for both low and high pressure. The low pressure is used on the press to take up the slack of the ram before the heavy pressure is applied. Then by simply turning a valve the maximum pressure of the accumulator is applied to the press, thus rendering its working very rapid and effecting a great saving of time; there is also a great saving in the matter of press cloth.

The pressure necessary to extract the oil has been the subject of much discussion. The best work, however, when the previous operations have been properly carried out, is obtained by applying a pressure of from 3,500 to 4,000 pounds per square inch. Some crushers apply 2,500 pounds, but this pressure, while extracting a large part of the oil from the crushed seed, leaves too great a percentage of oil in the pressed cakes; and, furthermore, requires more time for the cake to remain under pressure to obtain the full yield of oil.

64. Camel's-Hair Press Cloth.—What is known as **camel's-hair press cloth** is a woven fabric made from camel's hair. With the use of hydraulic presses and the accompanying enormous pressure to which the press-cloth material is subjected, camel's-hair cloth has been found suitable for good and economical working in the extraction of oil from cottonseed. The fine, long camel's hair, tenacious and elastic, is woven into cloth, resembling in appearance ordinary but very closely woven bagging, but it is capable of withstanding a pressure of from 3,500 to 4,000 pounds per square inch without being damaged.

A press cloth of this material, if used with intelligence and properly prepared meal only is pressed, will last a whole season. But when meal containing an excessive amount of moisture is placed in these cloths, they are more than likely to be ruptured and burst at the moment the heavy pressure is applied. With improper cooking and pressing of cottonseed meal, no matter how perfect the press-cloth material may be, its usefulness will be very rapidly destroyed. Many times a new press cloth has been rendered useless for further pressing during the first few charges of the press, owing to

these conditions. A press cloth made of good, long, staple camel's hair will last, when properly used, at least 6 weeks, the mill running the usual 24 hours, before it is rendered unserviceable.

65. Wool and Hair Press Cloths.—Press cloths made of **wool** also give excellent results in pressing cottonseed meal, but as a rule their high cost has prevented them from being used in the mills of the United States. In England and Egypt, however, wool press cloth is commonly used for pressing cottonseed.

In recent years, press cloths made of **horse hair** and **cow hair** have been found to give good results. On account of lasting so much longer under high pressure, they are said to be cheaper per ton of seed worked than either camel's hair or wool press cloths.

66. Cost of Press Cloths.—The item of expense for press cloth is always heavy in cottonseed milling, as the initial cost of the cloth itself is considerable. It is difficult to give any average cost, per ton of seed milled, for this material, owing to differences in manipulation. While one mill may crush for an entire season at a cost for press cloth of 6 cents per ton of seed, another mill in the same locality may have the cost per ton as high as 25 cents. In most cases, where such an excessive cost as the latter obtains, it is indicative of careless working. Where the cost per ton of seed crushed exceeds this figure, there is something radically wrong with the operations of the mill, probably with the cooking or with the pressing. A reasonable figure for press cloth under ordinary conditions should not exceed from 12 to 15 cents per ton of seed worked. Many successful mills work a whole season with an average cost for press cloth of about 10 cents per ton of cottonseed crushed.

The worst damage occurs to press cloth when poorly crushed seed, very hot material, and watery cooked meal are pressed. Any or all of these conditions exercise a very great influence on the life of the press cloth.

TREATMENT OF COTTONSEED CAKE

67. Preliminary Treatment.—When taken from the press and stripped, the cakes are very hard and solid, like a piece of board. Ordinarily, they are 14 in. × 32 in., which is the regulation size of cottonseed cakes. They are piled on trucks and sent to the cooling room, where they are piled on racks and cooled as rapidly as possible. A further purpose of this procedure is to give the material time to dry out, the evaporation of superfluous moisture being necessary

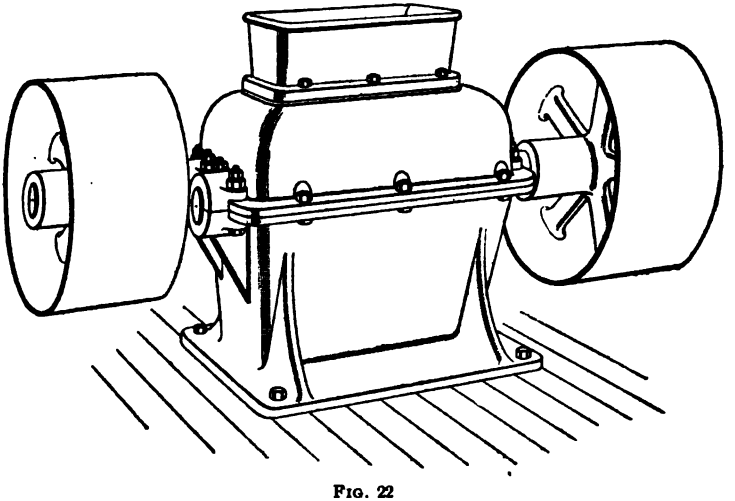


FIG. 22

to prevent the cakes from souring and molding. After remaining until cool, which requires from 12 to 24 hours, the cakes are fed into a machine known as a *cake breaker*.

68. Breaking the Cake.—The cake breaker, Fig. 22, revolves at a speed of 300 to 350 revolutions per minute. The cakes are fed into the cake breaker as fast as the machine will receive them and are broken into pieces of about the size of a chestnut, so that they may be conveyed mechanically to the mill for grinding into meal.

There are many forms of cake breakers, and many machines constructed for the purpose have failed to answer the rigid

requirements that this piece of apparatus must meet. The cake breaker must be very strongly constructed and be made so that soft and broken cake will not choke it. The cake breaker can be run with one belt where there is sufficient power.

69. Grinding the Cake Into Meal.—The broken cakes are conveyed from the breaker to **burrstone, or attrition, grinding mills.** Here they are ground into cottonseed meal, which is then placed in sacks, ready to be sold for stock-feeding purposes or to be used for fertilizer, etc.

In some cases, the ragged and uneven edges of the cakes are trimmed off and the trimmed cakes are packed in bags for export, some foreign merchants preferring the material in this condition, so that they can do their own grinding when desired.

MANUFACTURE OF UNDECORTICATED COTTONSEED CAKE

70. While the manufacture of **undecorticated cottonseed cake** is carried on to a very limited extent in the United States, this kind of cake is almost universally produced in England and Egypt from crushed cottonseed. The cottonseed from which the undecorticated cake is made, such as Sea Island and Egyptian seed, is usually free from adhering lint. The cottonseed as it comes from the gins may or may not be passed through the delinters to obtain the loose cotton accompanying it. Usually, without preliminary cleaning, the entire seed is passed directly to the upright serpentine crushing rolls and crushed as much as possible. The crushed seed is then directed to a large, flat pan that holds as much material as the presses. In the pan are two large, heavy, circular, upright stones, known technically as *edge runners.* These revolving stones weigh several tons and are kept running over the seed in the pan with a circulatory motion until the seed is reduced to the required fineness for heating and subsequent pressing.

71. After the cottonseed is in the required condition for pressing, the material is transferred to cooking kettles that are similar in construction to the usual American kettles, but have doors on the lower edge from which spouts lead downwards. The crushed seed is cooked in the kettles until ready for pressing, when the material is filled into bags made of woolen press cloth and placed in the hydraulic press; here, the crude cottonseed oil is pressed out in the usual manner. The more modern method practiced on undecorticated seed follows closely the American method of filling the cooked seed into a cake former and then wrapping it with the usual kind of press cloth.

The presses usually employed for pressing are unlike the American presses in that they have open sides. The presses have this form because the crushed whole seed containing all the hulls holds together sufficiently well to have the oil extracted by the usual hydraulic pressure, and keep the form of cake without any difficulty. The box-plate, or enclosed, press plate is gradually replacing the open-side presses used in the English mills, as it has been found that they are more economical in working and give better results.

The undecorticated cottonseed cakes are removed from the press and the press cloths are stripped from them. After this they are trimmed on all the edges to remove the parts that contain too much oil. The trimming of the cakes is done by a cake parer, the oily edges that are removed being returned to a new portion of seed being ground under the edge runners. Any broken pieces of cake are also returned to the edge-runner pan and mixed with the fresh material being prepared for pressing.

72. After the cakes are pared, they are placed in racks for cooling, this being a necessary precaution to prevent heating and molding. When thoroughly cooled, the cakes are usually piled in tiers, as their nature and physical characteristics are such that they are always shipped loose, even to foreign countries, without being bagged or protected in any way.

Undecorticated cake is dark, usually greenish or brownish in color, is hard and naturally very rough in texture, and shows throughout large pieces of the hulls. The undecorticated cake manufactured in the United States is used only for fertilizer purposes, while in England and other countries it is highly valued as a cattle food.

Undecorticated cake as made in English mills usually contains about 9 or 10 per cent. of moisture, from 4.5 to 5.5 per cent. of oil, and from 22.5 to 24.5 per cent. of protein.

73. The crude cottonseed oil obtained from the pressing of the crushed whole seed is usually very dark in color and has a strong odor, owing to the fact that the hulls and kernels are cooked together. Crude cottonseed oil obtained by the undecorticated method of manufacture will never refine to the high quality of American cottonseed oils, as is evident from the different methods of manufacture. As a rule, crude oils made by the English method contain a high percentage of free fatty acids, owing to the age of the seed usually crushed, the long time that elapses during transportation before arriving at the mills, and the quantity of cracked and broken seed mixed with the usual shipments of Egyptian cottonseed, caused by the many rehandlings between the source of the supply and the point of use.

74. In several English mills, Indian cottonseed, or, as it is termed in the trade, "Bombay" seed, is crushed in the manner just described; but it is not a popular cake, because the large amount of lint present in the undecorticated cottonseed cake is objectionable.

Mixtures of Indian cottonseed and Egyptian seed are also sometimes worked together to make an undecorticated cottonseed cake when the difference in price between the two is considerable.

English undecorticated cottonseed cakes are not ground into cottonseed meal for feeding purposes, but are simply broken into pieces by the usual cake breaker and fed to cattle in that form.

COTTONSEED OIL AND PRODUCTS

(PART 2)

REFINING OF CRUDE COTTONSEED OIL

INTRODUCTION

1. As previously noted, the condition of the seed influences to a great extent the quality of the crude oil produced. When cottonseed has been stored for any length of time and becomes heated, owing to the presence of moisture, the free fatty acids resulting from decomposition will appear to a marked degree in the oil made from such seed. The greater the quantity of free fatty acids in the oil the less will be its market value, the greater the shrinkage in refining, and the lower the grade of the finished product.

Crude cottonseed oil as obtained from the oil press is a thick fluid, varying in color from yellowish brown to a dark ruby or blackish red, according to the nature and condition of the seed from which it has been pressed. It contains much moisture or water, and impurities consisting of fine meal, "mealy matter," coloring matter termed *Gossypium*, mucilage, and more or less tarry matter. Most of these impurities are due to the application of heat and moisture in pressing the oil from the cooked seed. If it were commercially profitable to press cottonseed by the cold process, an almost white oil could be obtained, in common with most vegetable seed oils. Cold pressing is carried on to a limited extent in France, but the price obtained for the product is commensurate with its advanced cost of manufacture. The

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yield by the cold-pressure system of extracting cottonseed oil is far below that from most other vegetable seeds, such as linseed and rape seed.

The yield of cottonseed oil by the usual cooking ranges from 12 per cent., with poor working and from poor seed, to 17 per cent.—the latter in exceptional cases and from good seed. The general average yield may be taken as 15 per cent., or 300 pounds of oil to the ton. The general trade requirements of crude oil are explicitly set forth later.

While the oil miller may not readily be able to dispose of his crude product, he is enabled by means of the oil refinery to convert it into oils of higher grade, and, if provided with the necessary equipment, into finished articles of which cottonseed oil is the base.

2. Crude oils containing less than 1 per cent. of free fatty acids are used in the manufacture of butter oils. Only selected seed is used, and great care is taken in the process of manufacture to avoid any unpleasant odor or taste.

Crude oils testing between 1 and 2 per cent. of free fatty acids are refined in the regular way to a prime yellow oil and should grade as such.

Crude oils containing more than 2 per cent. of free fatty acids are unsuitable for edible oils, as their original reddish-yellow color does not disappear on refining.

Crude oil may have as high as 7 or 8 per cent. of free fatty acids in exceptional cases, but where seed has become badly damaged by heating, etc., this is not uncommon. Such crude oil should be handled to produce as high a grade as possible with a minimum amount of loss in refining. Oil of this nature is suitable, naturally, only for industrial and not for food purposes.

The loss in the refining of crude cottonseed oils varies. The greater the amount of free fatty acids, the greater will be the loss in refining. The average loss in the refining of good cottonseed oils approximates from 8 to 10 per cent. In many cases, this loss is much less, and, likewise, in many other cases, it is much greater.

APPARATUS AND METHODS USED IN REFINING CRUDE COTTONSEED OIL

3. The real advancement of the last 25 years in the cottonseed-oil industry has been made in the oil refinery. While there have been many improvements in the machinery of the crude-oil mills, the process is today practically what it was a quarter of a century ago; but tremendous strides have been made in the improvement of the refining methods, and the products obtained at this time are quite superior to those formerly produced.

APPARATUS FOR REFINING

4. The methods used in the United States for the clarification, or refining, of cottonseed oil vary considerably in the details, but all are based on the use of a caustic alkali, employed either alone or with certain adjuncts. The alkali almost universally used is caustic soda, and the adjuncts used in connection with refining are permanganate or bichromate of potash, and sulphuric or hydrochloric acid, chlorine gas, or oxygen. While caustic soda in solution is now almost exclusively the only reagent used in the largest refineries, at times this is supplemented by other agencies.

The apparatus necessary for refining is shown diagrammatically in Fig. 1. It consists of a lye tank *A* for holding the solution of caustic soda, a refining tank *C*, a soap-stock tank *D*, and a finishing, or rectifying, tank *E*. The lye tank is placed some distance above the refining tank. The former is made of boiler iron and hence is not subject to leakage. The tank is fitted with $1\frac{1}{2}$-inch pipes that are controlled by a cock for regulating the supply of caustic soda. A perforated sheet of iron, or a sieve, *b* is placed under these pipes and over the refining kettle, covering about three-quarters of its surface. This permits the lye to be evenly and finely distributed over the surface of the crude oil and insures a thorough, intimate mixing of oil and lye by agitation, which is a most essential part of the operation. The lye is in many

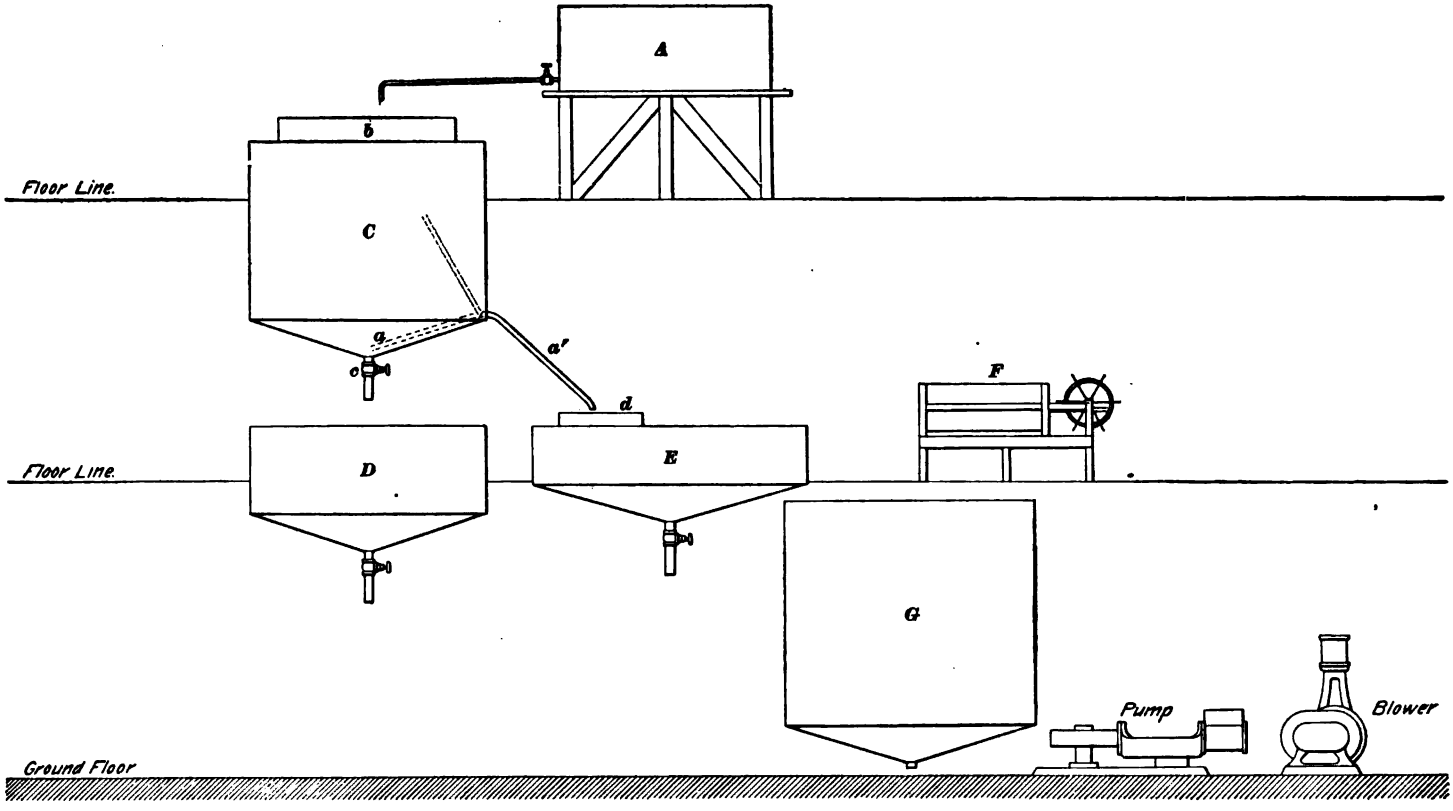

A
b
Floor Line.
C
a
a'
c
d
F
Floor Line.
D
E
G
Pump
Blower
Ground Floor

FIG. 1

cases added to the crude oil by means of a perforated pipe extending across the refining tank. This is the most modern method of adding the lye to the crude oil.

5. Refining Tank and Its Accessories.—The **refining tank** is circular in shape, of sheet iron, and of a size adapted to the requirements of the plant. It is usually made to hold from 7,000 to 12,000 gallons. The diameter is about one-third greater than the depth. There are several methods of agitating the oil while it is under treatment; the principal ones consist of agitation by the *air blower*, by means of the *paddle wheel*, or by the *screw propeller*.

6. The **air pump,** or **blower,** is extensively used, but it has been discarded by some refiners using the caustic soda alone, because the heavier caustic solution resists much more than the lighter oil the agitation from a stream of air forced into the mixture by the blower. This having been fully demonstrated, it is obvious that there is imperfect agitation and mixing, and a consequent loss in greater shrinkage by the use of this method. A further objection to air agitation is the greater time required for the finely divided foots to settle.

7. The **paddle wheel,** which is similar in style and shape to the ordinary side paddle wheel of a steamer, is made so that it will just about fit the refining tank. It is actuated by a pulley on an extension of its shaft, which extends through one of the sides of the tank. When the wheel is set in motion, it stirs the oil and caustic solution and effects a thorough mixture.

8. The **screw propeller** is like the ordinary type of ship propeller, with a pitch designed to throw up the oil at an angle of a little less than 45°. It is placed at the bottom of the tank and is driven from above, its shaft descending into the tank. The propeller need not be over one-third the diameter of the tank, because, when in action, it creates a motion that effectually agitates the oil and mixes it intimately with the caustic solution.

9. The refining tank is provided with closed steam coils 1½ inches in diameter. These pipes are placed at the bottom of the tank and continue up along the sides to within two-thirds of the top. They serve to supply the necessary heat to the oil when ready to work. In addition to these, open steam pipes are placed in the bottom of the tanks, as live steam is helpful in refining. The bottom of the tank is made slightly cone-shaped so as to facilitate the discharge of the stock by means of a large cock placed in the center, or apex, of the bottom. The refining tank is placed just above the soap-stock tank, so as to discharge the formed foots by gravity.

10. When air is used for agitation, the same pipes that inject air may also be made available to inject live steam by simply making the necessary connections outside the tank. When this is to be done, the pipe conveying air from the compressor into the oil should enter, with as few sharp turns as possible, the center of the tank at the top and descend vertically therein to the bottom. The main air pipe should be at least 4 inches in diameter, 6 inches being still better for this purpose. From the end of this pipe a number of 1-inch pipes radiate. These small pipes extend along the bottom of the tank and are about one-third the diameter of the tank in length. Near the under side and at an angle of 45°, they are perforated with holes $\frac{1}{16}$ inch in diameter so as to give a scouring force to the jets of air. It is a most essential point that the area of the openings in the small pipes should not exceed that of the main supply pipe, otherwise the blower will not do effective work. The ends of the pipes are capped, with a perforation in each cap. The appearance of the arrangement of pipes is similar to that of a spoked wheel without the tire. Arranged in this manner, the air from the pipes creates an upheaval of oil in the center. The entire mass, in other words, flows up in the center and down at the sides, the operation thus causing a motion from the center to the circumference. In this way, the maximum utility of the air is obtained.

11. Soap-Stock Tank.—The **soap-stock tank** is placed directly beneath the refining tank. It is circular in shape, is made of sheet iron, and is provided with a closed steam coil. It usually has the same diameter as the refining tank, but is only half the depth. The bottom is also made similar to that of the refining tank and has closed steam pipes, the purpose of which will be explained later.

12. Finishing, or Rectifying, Tank.—The **finishing, or rectifying, tank** is a large, flat tank provided with closed steam coils $1\frac{1}{2}$ inches in diameter and with perforated air pipes. Its depth should not exceed 3 feet and that of the oil placed therein $2\frac{1}{2}$ feet. The tank is usually made large enough to hold the oil from one treatment of crude oil.

METHODS OF REFINING

13. The general arrangement of a refinery is shown in Fig. 1. The apparatus just described, together with the air blower, pump, and necessary power, is practically all that is required for the refining of crude cottonseed oil. The size of the tanks, the arrangements, and general detail must naturally be governed by circumstances in each place where oil is refined. The general operation is carried out in the following manner, more specific details being discussed in later pages.

14. Refining Crude Oil by the Caustic-Soda Method. In the **caustic-soda method,** the crude oil is heated in the refining tank *C*, Fig. 1, to about 80° F., sufficient room being left in the tank for the solution of alkali and for gentle agitation. The agitator is set in motion, and, when fairly started, is regulated so as to give the oil a slow and regular rolling motion. The cock on the caustic-soda tank shown at *A* is then opened, and the lye is allowed to drop slowly and gradually through the screen *b* or through a perforated pipe into the oil in the tank *C*. The quantity of lye necessary depends wholly on the quality of the crude oil, from 5 to 15 per cent. of the volume of the oil generally being

used. The quantity is also governed by the strength of the lye used for refining purposes.

During the mixing of the lye with the oil, the operation is closely watched to ascertain when sufficient alkali has been added. The appearance of the oil changes in a few minutes after the introduction of the soda solution. The bright-yellow froth, always present on the oil after agitation through pumping, etc., gradually disappears and that remaining has a tendency to bleach. The oil assumes a brownish hue, becomes more fluid, and gradually turns blackish in color. This change will take place about 15 or 20 minutes after the operation is begun. When a sample withdrawn in a ladle shows a clear, bright oil intermixed with small blackish clots, the critical moment has been reached; the experienced operator always relies on his eye to judge the "turning," or "breaking," in the oil. The ordinary and usual method is to dip with the hand a portion of the oil, when the separation in the palm is easily observed. When this condition is observed, the flow of the caustic-soda solution is immediately shut off and the agitation reduced to maintain a long, rolling motion. The oil is now heated; at a temperature between 100° and 120° F. the oil will break well and the agitation will cause the agglomeration of the particles of soap stock, so that the oil will settle clear. The time required for the refining operation is from 30 to 45 minutes.

15. During the process of refining, the oil may be judged as to the degree of refining in the following way: A portion of the oil while agitating in the tank is withdrawn and filtered into an ordinary sample bottle; the color of the filtered oil may be compared with a standard refined-oil sample to see if the refining is complete. A few tests of this description, when the oil appears to be refined, will show the operator how much further to proceed and, approximately, how much more lye should be added to produce the required yellow oil. Practical experience, combined with the knowledge of conditions existing at the time, together with the aid of chemistry in determining how much lye is required to

neutralize the free fatty acids in the crude oil, very shortly enables the operator to make quick and perfect refinings.

16. When the oil is seen to be settling well in the refining tank, the agitation is wholly stopped and the refined oil allowed to settle. It is the general practice to allow the oil to settle overnight, although in some refineries the supernatant oil is withdrawn after settling for 4 or 5 hours. In treating prime crude oil, the temperature in the refining tank must not exceed 120° F. As a general rule, the best results are obtained in refining the oils at the lowest practicable temperature. In every case, the temperature should be kept as low as possible while refining, especially with crude oils of good quality. In any case, a high degree of heat has an injurious effect on the flavor of the better grades of oils.

17. The clear oil is drawn off from the tank by means of the siphon *a*, Fig. 1, through the pipe *a'*, into the finishing tank *E*. The screen *d*, covered with gauze or cheese cloth, serves to catch and retain any fine particles of soap stock that may be carried over when the siphon is withdrawing from the refining tank the oil just above the settled soap stock.

In the finishing tank, the oil is heated to 120° F. and blown with air until a sample, withdrawn in the tester or sample bottle, remains clear and unclouded when chilled to 60° F. When this condition is observed, the oil is free from moisture and is then allowed to settle or is pumped through the filter press *F* into the storage tank *G*. It may be barreled when cool as summer-yellow cottonseed oil.

18. While the foregoing is the general plan followed, many of the details in some mills are worked out in a different manner after the crude oil is settled. For example, in some refineries, after the oil is sufficiently refined, it is treated with a weak brine that is made by dissolving 1 per cent. of salt in hot water. This is agitated with the freshly refined oil, which procedure materially assists in the clarification. After withdrawing the yellow oil, some refiners subject it to a thorough washing with fresh water to remove

all possible traces of alkali. This course is followed particularly with oils desired for edible uses.

By means of the filter press, the operation of settling or removing the impurities may be materially shortened; also, other advantages that will be described later will be gained.

19. Refining Crude Oil by the Caustic-Soda, Bichromate-of-Potash, and Sulphuric-Acid Process. As has been stated, the caustic-soda process is sometimes used with other adjuncts in the process of refining crude oils. With oil extracted from heated seed, or seed otherwise unsound, the use of caustic soda alone is not capable of producing a bright-yellow oil from the crude article. Sometimes, therefore, it is necessary to supplement the effects of caustic soda by those produced by other and more potent agencies. The method generally practiced is the following:

20. The crude oil is given the preliminary treatment with the caustic-soda solution, but as soon as it is thoroughly cleaned and is of an off-yellow, or reddish, cast, it is transferred into another clarifier and there thoroughly washed with free superheated steam. This serves the double purpose of cleaning and bleaching the oil. The oil is then allowed to settle, after which it is drawn into a lead-lined tank for subsequent treatment. The lead-lined tank is provided with copper steam coils, and agitation is effected by means of the air pump and perforated lead pipes that are arranged as in the ordinary refining tank. The oil, having been placed in this tank, is slowly heated to 100° F.

A 10-per-cent. solution of bichromate of potash having been prepared with filtered water, a quantity of the solution, equal in volume to from $2\frac{1}{2}$ to 5 per cent. of the oil, is sprinkled over the surface of the oil while it is being agitated. As soon as the oil appears of a dark-green or even greenish tinge, the supply of bichromate solution is stopped. The agitation is continued and chemically pure sulphuric acid, diluted one-half or three-quarters with water, is slowly added to the oil. The condition of the oil is tested from time to time by means of the test glass. When the

precipitation is complete, as may be easily seen in the glass, the supply of acid is stopped, the air agitation shut off, and the mixture allowed to rest for a few hours.

21. The underlying acid water is withdrawn from the bottom, and the oil is washed with warm water until all traces of the acid have disappeared. The material is then sent to the rectifying tank, as in the ordinary caustic-soda process. Sometimes, previous to washing the oil with water, a washing with weak lime water is resorted to, but poor as the oil may be, this operation is injurious to the flavor of the finished product. Consequently, it is used only in the case of very poor oils. Ordinarily, the best method is to use only warm water in a tank with paddles, which give the oil a thorough beating and washing. This necessitates the decanting of the oil into one of the iron clarifying tanks. Air agitation may be used for beating and washing, but the other method is considered more reliable and effective. If it is the intention to make from such crude oil a white oil, the refining in the first instance with the caustic soda is pushed to the straw color of prime refined cottonseed oil. The oil should then be steamed and finally treated as just described. This obviates the necessity of using an excessive amount of acid.

22. Refining Crude Oil by the Permanganate-of-Potash Method.—Caustic soda, permanganate of potash, and sulphuric or hydrochloric acid are used in the **permanganate-of-potash process.** With some oils, permanganate of potash gives better results than bichromate, and enables the refiner to make a palatable oil out of an inferior article, otherwise suitable for crude industrial purposes only. The same may be said of sulphuric and hydrochloric acids used in connection with the permanganate. With certain oils, one does better work than the other, and nothing short of an actual laboratory test can guide the operator in deciding whether to use bichromate or permanganate of potash. Such tests should be made on all oils of an inferior grade before attempting to work with the usual large quantities.

The method of treating the oil by the permanganate process is carried out in the same manner as has been described. The proportions of the ingredients used are also the same as in the bichromate method.

The bleaching of cottonseed oil by means of chlorine gas or oxygen is not done in many of the refineries of the United States, the caustic-soda method being employed in by far the greater number of refineries as a preliminary to the bleaching of a yellow oil.

23. Refining or Bleaching Crude Oil by Ocher. Among the various methods of bleaching cottonseed oil is a patented process in which the use of an iron ore, such as limonite, hematite, or ocher, is used. With some oils, it does excellent work, but the fact that the process is protected by patent probably operates to prevent its more general adoption. This method, besides being applicable to cottonseed oil, is also used with great success for the refining of linseed, rape seed, and similar vegetable oils.

The process is carried out by the use of the substances just mentioned or of any similar substance of the nature of a pigment that does not injure the oil as an article of food. By mixing and agitating the ocher, which has been previously prepared, with the crude oil to be refined, the mucilage, gum, and coloring matters are absorbed by the ocher, which settles to the bottom with the impurities, leaving the supernatant oil clear and refined. The refining agent is first prepared by heating, so as to drive off all moisture, which is an essential part of the operation. In some cases, the ocher is dried and finely pulverized and used in the proportion of 12 parts of oil to 1 part of ocher. The combined oil and ocher is then briskly agitated for about 35 or 40 minutes, when the refining will be completed. Naturally, these proportions, as with other methods of working, vary with the amount of impurities the oil contains.

The oil is separated from the ocher by the use of the filter press or is allowed to remain in the bleaching tank for 24 hours. In the latter case, the clear supernatant oil is

drawn off, leaving only the settlings to be filtered. When removed from the filter press, these settlings make a stiff paste consisting of ocher mixed with all the impurities and a small amount of oil.

24. The inventor of the ocher process of refining further claims that by a certain combination of refining or purifying agents, rancid oil may be successfully reclaimed by his process. For oils that have undergone more or less change by oxidation and are heavily charged with free fatty acids and other impurities, the ocher is reduced to a fine powder and 10 per cent. of the amount to be used is mixed with finely powdered caustic soda or borax, the amount used being equal to from .5 to 1.5 per cent. of the weight of oil with which it is mixed. This mixture is then added to the oil to be refined and the whole agitated thoroughly for 10 or 15 minutes. After this period, a mixture of salt or calcium chloride in saturated solution, equal to twice the weight of the first mixture, is added to the oil and the agitation continued for 10 or 15 minutes longer. The remaining portion of the ocher necessary to complete the refining is now added to the mixture and the agitation continued for a like period, the temperature of the oil being maintained at about 90° F. The oil is then passed through the filter press to remove the refining agents and the impurities. The chief objection to the use of this combination process, or to the use of ocher itself, is the shrinkage in refining, owing to the necessary saturation of the ocher with the oil. But with rancid oils the process might be made very serviceable.

25. Special Treatments and Precautions in Crude-Oil Refining.—In the refining of crude cottonseed oils, many of the details, which are largely influenced by the desired character and final use of the refined oil, vary in different refineries. Oils that are intended for food purposes cannot, or, more truly, should not, be treated as oils intended for industrial purposes. For the former purposes, as low a temperature as practicable should be given such oils, as the whole object is to make them as neutral and as bland as

possible, with no rank or strong odor, which is always produced in oils when a high temperature is used in working. In the finishing tank, care must be taken to keep the oil intended for edible purposes at as low a temperature as is compatible with finishing the product. This will be more fully described under the heading Butter Oil.

26. An oil unduly heated in the initial process of refining invarably requires more heat in the last processes to brighten it, and such heat is fatal to its essential characteristics as a table oil.

When cottonseed oils are refined for industrial purposes, before the caustic-soda solution is dropped into them, they may be heated as high as 110° F. After the cold lye has been added and the proper breaking of the oil observed, the supply of caustic solution is shut off and the mass in the refining kettle heated, by means of the closed steam pipes, to a temperature of 130° F., the agitation being continued during this heating. At this temperature the steam is shut off, the agitation stopped, and the oil allowed to settle as usual. It is then drawn into the rectifying, or refining, tank and heated in order to impart to the oil the necessary brightness. This oil may be heated to 250° or 285° F., but no higher. By this method the loss in refining the oil is decreased, but the oil is of inferior flavor and taste. In other respects, it is similar to oils refined at the lower temperatures.

27. Steaming the oil is resorted to in some refineries both before and after clarification. For steaming purposes, the oil is pumped through the filter press into another clarifier and free steam is injected into the oil. In order to obviate too great condensation of steam in the oil, the latter is at the same time heated by the closed steam coils. As live steam does not injure oil, the heat may be raised without fear of bad results to 280° or 290° F. The steaming process not only has the advantage of washing the oil, but at the same time the oil is bleached, first, by the oxygen injected with the steam and, second, by forcing out of it the

coloring matter that is held only in mechanical suspension by the oil; after the process of steaming is finished, this suspended matter, together with the water, settles to the bottom of the tank.

The steaming process must not be carried on for too long a time, as oil so treated has a tendency to oxidize more or less and become strong and rancid smelling. But, properly handled, steaming for from 10 to 15 minutes generally improves poor-grade oils. No hard-and-fast rules can be laid down as to the time of steaming, as this depends on the quality of the oil, and practical experience is the only guide that can be safely followed. Another advantage is the fact that the entrance of the caustic-soda solution may be stopped when the oil is yet reddish in color and the remaining color driven out by steam. The steaming also effectually removes all traces of alkali that may remain after the caustic-soda treatment. After being steamed, the oil goes to the rectifier for drying out, as usual.

The steaming of crude cottonseed oil prior to clarification is resorted to more particularly with inferior or unclean oils. The free steam dissolves the mucilage of the oil and facilitates its precipitation, as well as many of the impurities that the oil gathers, especially from tanks and barrels, when received in such receptacles. In many instances, oils are washed with warm water after clarification and are afterwards drawn off into the rectifying, or finishing, tank. Off-cottonseed oils may frequently be improved and more or less deodorized by treatment with live steam in the requisite tank.

28. Refining Cottonseed Oil by Brine and Caustic Soda.—A process of refining cottonseed oil by means of brine and caustic soda, for which a patent was issued a few years ago, is said to be very satisfactory. It consists in agitating the crude cottonseed oil in a tank in which is mixed a suitable proportion of salt water. When a thorough mixture is obtained, the usual caustic-soda solution is added until the oil is broken. The agitation is then stopped and warm water is sprinkled over the oil. The clear oil rises to

the top, the coloring matter subsiding with the water. The temperature during the operation is maintained at 80° F.

29. Loss, or Shrinkage, in Refining.—The loss, or shrinkage, in refining oil depends wholly on the grade of the crude oil operated on. While the usual loss in refining will average about 8 or 10 per cent. in the United States, it may fall as low as 3 per cent., as has been repeatedly demonstrated. Again, with a very poor oil, the shrinkage will reach as high as 15 per cent. or more, but the latter figure is rather unusual with the average grade of crude oils. The oils from different sections of the South, while apparently of the same grade, produce very different results as to the loss in the usual methods of refining. The quality of oil also varies in different seasons from seed obtained from the same sources.

Each batch of crude oil to be refined presents, in a way, an original proposition, previous experience with other oils being no help or guide as directly applied to the lot under consideration. Individual lots must have applied to them individual treatment. For this purpose, the color test, described later, is determined for each separate batch of crude oil and the treatment determined from the data obtained from the test.

30. Cottonseed Foots and Their Treatment.—After withdrawing all the oil obtainable from the refining tank, there remains in the bottom a light brownish material consisting of soap, mucilaginous matter, coloring matter, and other impurities mixed with more or less of the refined cottonseed oil. This material is technically known as **cottonseed foots**, and in many instances is also known as **soap stock.**

The foots are withdrawn into the soap-stock tank *D*, Fig. 1, immediately below the refining kettle, through the large valve *c*. They are then slowly heated by means of the closed steam coil in the kettle and the oil is removed, as far as practicable, and mixed with the crude oil for subsequent refining operations. The stock is then cooked and

barreled while in a fluid condition and is sold under the name of cottonseed foots to soap manufacturers on the basis of the contained fatty acids.

In many establishments, the foots are made into soap stock, and subsequently soap, without the intermediate expense of barreling. When fresh, cottonseed foots are of a brownish color, but they rapidly become black on exposure to the air. In some plants, a crude system of pressing is resorted to for obtaining all the oil possible from the foots. This is an economical procedure, as there is always a wide difference in value between the foots and the oil. By the usual procedure, the free oil remaining in the foots may be saponified, and thus a homogeneous soap obtained. This is known in the trade as **cottonseed soap stock.**

31. The foots obtained by refining may be still further manipulated, if desired, by resolving them into fatty acids, water, and impurities. For this purpose, the foots are brought to a warm liquid state with water; dilute sulphuric acid is then added to the solution, and the whole solution boiled with live steam. The liberated fatty acids formed from the decomposed soap rise to the top in a distinct layer when the material is allowed a period of rest, and may be removed in a more or less pure condition by simply withdrawing the underlying acid water. Sufficient acids must be added to foots to decompose wholly the alkaline matters present. The operation may be carried on in a wooden vat, but is preferably performed in a lead-lined receptacle. The fatty acids obtained are technically known, in many instances, as **black grease.**

This material may be still further manipulated by being distilled with superheated steam into stearin (of the grease) and cottonseed pitch, the former distilling over and the latter remaining behind in the still. This operation is practiced more extensively in England, France, and Germany than in the United States. The stearin obtained in this way must not be confounded with the cottonseed stearin obtained by cold-pressing summer oils.

32. Preparation of the Caustic-Soda Solution for Refining.—The dissolving of caustic soda used in the refining is a simple operation, yet much time and annoyance may be saved by having the lye tank properly constructed. If elevated above the level of the floor, it should be conveniently near the refining tank and at a higher level so as to allow the solution to flow into the oil by gravity.

The lye tank should be provided with a perforated-metal false bottom to support the broken lumps of caustic soda. This greatly facilitates the dissolving process by causing a natural circulation of the liquors of different densities, and by this, fresh, lighter liquors are constantly dissolving the caustic. The saturated liquor, owing to its greater density, sinks to the bottom of the tank. Where the false bottom is not in the lye tank, mechanical agitation must be resorted to for dissolving the alkali rapidly.

33. If care is not taken to agitate the material when placing caustic soda directly in the lye tank, the soda, owing to the heat generated by its combination with water, will melt and form a thick, solid layer on the bottom of the tank. This layer after becoming cold will be very difficult to dissolve. Another disadvantage of allowing the caustic soda to dissolve slowly is that it is very liable to become carbonated, thus reducing its effectiveness in refining where a full caustic effect is required. A carbonated caustic-soda solution is an uncertain factor with which to work in refining. The solution of alkali should always be kept tightly covered and kept at the degree of strength best adapted for refining the usual run of oils; the degree of strength usually selected is 20° Baumé for the stock tank, but of course varies with circumstances.

In some refineries, the stock of lye in the tank is kept in any convenient place, and the amount of solution required for each batch of oil is pumped from the stock tank into the elevated lye tank above the refining kettle. In many refineries the dissolving tank is located on the ground floor, which is a very convenient arrangement.

34. Caustic soda comes to market in a great variety of grades and is always sold on a basis of 60 per cent. of sodium oxide, Na_2O. The common impurities occurring in caustic soda are sodium chloride, sodium carbonate, and sodium sulphate. It is of great importance to the refiner to know the actual caustic strength of the soda and the amount of impurities in it. In the preparation of the lye, the salts referred to reduce the active value of the solution for the indicated specific gravity or degrees Baumé. In other words, a lye of 20° Baumé, containing a considerable amount of impurities in the form of carbonate, etc., would not perform the work in refining the oil that a lye of the same degrees Baumé prepared from a caustic soda of high grade, free from any considerable amount of these salts, would do.

As the reduction in the active value (saponifying power) is least in the highest grades and greatest in the lower grades, as a result of the larger proportion of impurities present, it is not only uneconomical to use a low grade of caustic soda in refining, but it often leads to uncertain results.

TABLE I

PERCENTAGE OF SODIUM HYDRATE, *NaOH*, IN LYES OF DIFFERENT DENSITIES MADE FROM CAUSTIC SODA OF VARIOUS GRADES

Specific Gravity	Degrees Baumé	Percentage of Sodium Hydrate, *NaOH*					
		77½	76	74	72	70	60
1.075	10	6.55	6.42	6.25	6.08	5.91	5.06
1.091	12	8.00	7.84	7.63	7.43	7.22	6.19
1.116	15	10.06	9.86	9.60	9.34	9.08	7.78
1.142	18	12.64	12.40	12.07	11.74	11.41	9.78
1.162	20	14.37	14.09	13.72	13.35	12.97	11.12
1.180	22	15.91	15.61	15.19	14.78	14.36	12.31
1.210	25	18.58	18.23	17.74	17.27	16.78	14.38
1.241	28	21.42	20.99	20.44	19.89	19.33	16.57
1.263	30	23.67	23.21	22.60	21.99	21.37	18.32
1.320	35	28.83	28.28	27.53	26.79	26.04	22.31

35. Table I shows to what extent an indicated Baumé reading may show a varying strength of actual caustic soda

present in a lye prepared from different grades of ordinary merchantable caustic soda. It is assumed that all the alkali present is entirely caustic soda, and while the results are perhaps slightly higher than would be found in actual practice, the table is of value and sufficiently accurate for technical purposes. The oil refiner may conveniently consult this table in preparing his lyes for refining crude oils.

36. For the reasons given, caustic sodas of 74 and 76 per cent. are superseding the lower grades in the refining of crude cottonseed oils. The caustic is best melted by a jet of steam until the solution is of a density of about 35° Baumé. The solution is then allowed to settle and, if necessary, is drawn off into another vat.

The stock-lye tank is usually kept at a strength of 20° Baumé, being reduced or strengthened as occasion demands. For making butter oil, the strength is reduced to 10° or 12° Baumé. For ordinary oils, a strength of 20° Baumé is used, while for poor and bad oils a strength of 25° Baumé may be used. The volume of lye used varies from 5 to 20 per cent. of that of the oil, according to the quality of the crude cottonseed oil.

REFINED COTTONSEED-OIL PRODUCTS

MANUFACTURE OF VARIOUS GRADES OF REFINED COTTONSEED OIL

37. Butter Oil.—The grade of oil known in the trade as **butter oil** is a very choice summer-yellow cottonseed oil made from selected seed, as a rule, and treated somewhat differently in refining from prime summer-yellow oil. Butter oil can usually be made only from crude oils testing not over 1 per cent. in free fatty acids. The crude product must be handled very carefully, with reference to the temperatures to which it is subjected, in every operation from the start to the finish. With this oil it is imperative to have every tank holding it scrupulously clean. Although cleanliness should prevail with every grade of oil, a high-grade butter oil cannot be obtained unless conditions of cleanliness, extending even to the barrels into which the oil is drawn for shipment, are rigidly maintained.

38. Butter oil must meet the trade requirements as to color, taste, flavor, etc., and the loss to the manufacturer in refining must be reduced to a minimum. The process of manufacture is carried out as described in Arts. **13** to **18**, with the following modifications: The crude oil selected for manipulation is freed as far as possible from the ever-present impurities by subsidence, decantation, or filtration. An oil that has been undisturbed for several days or weeks usually has all mealy matter, mucilage, etc. wholly settled from it, and, being free from sediment, it may be pumped directly to the refining tank. Where rapid manipulation of freshly made crude oil is desired, recourse must be had to the filter press to eliminate the impurities.

39. The crude oil is warmed to 75° F. and the agitator started as usual. The caustic-soda solution for refining should be reduced to a strength of about 10° Baumé and in no case should it exceed 12° Baumé. The oil is agitated in the usual manner and the lye added, as previously described. Care should be taken not to heat this crude oil in the refining tank above 105° F. After the oil has been refined and well settled in the usual manner, it is drawn off from the foots into a thoroughly clean tank and there washed free from all traces of alkali. The butter oil is then freed from moisture by heating and blowing, the temperature in the finishing tank in no case exceeding 120° F., after which the oil is pumped through the filter press to clear and brighten it. The filtered oil, which should be of a light lemon-yellow color and free from all undesirable odor and flavor, is sent to the storage tank for barreling after it has regained its normal temperature. Butter oil should be as neutral as possible in all its characteristics.

40. Yellow Cottonseed Oil.—The grade known as **yellow cottonseed oil** is the staple cottonseed product obtained by refining the crude oil. It is sometimes heated in a jacketed tank to remove any strong odor and flavor that may be present in a marked degree. The dried and filtered oil is slowly heated to a temperature of 370° or 400° F. and, in some cases, very lightly blown for 3 or 4 hours. This treatment is very beneficial to some oils, but it is disadvantageous to others. Frequently, the yellow oil is simply heated to 400° F. and held at that temperature for 4 hours, when most of the strong and persistent odor of the cottonseed will be eliminated. Oils treated in this way are liable to have fixed in them the yellow coloring matter, rendering them incapable of being made into a water-white cottonseed oil. Such oil, however, is suitable for use in articles in which their yellow color is not objectionable, such as in oleomargarine, "cotto" compounds, etc. Care must be taken not to blow the oil too strongly or for too long a period.

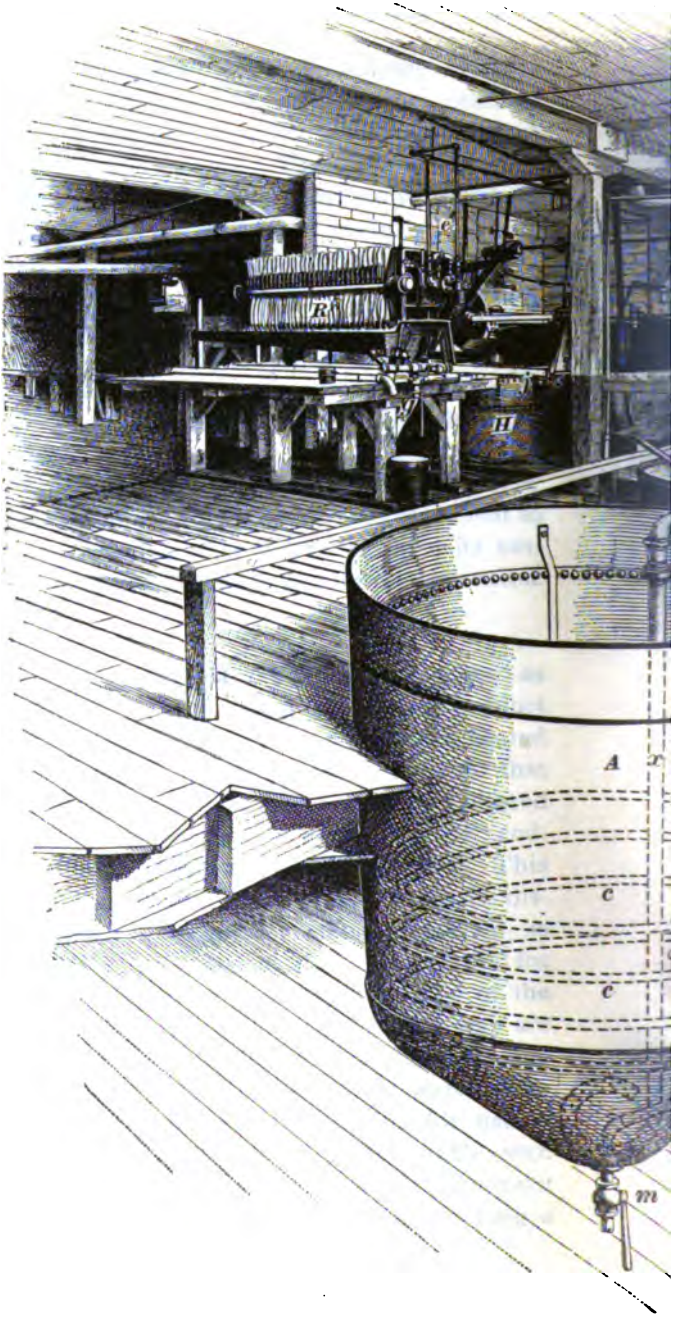
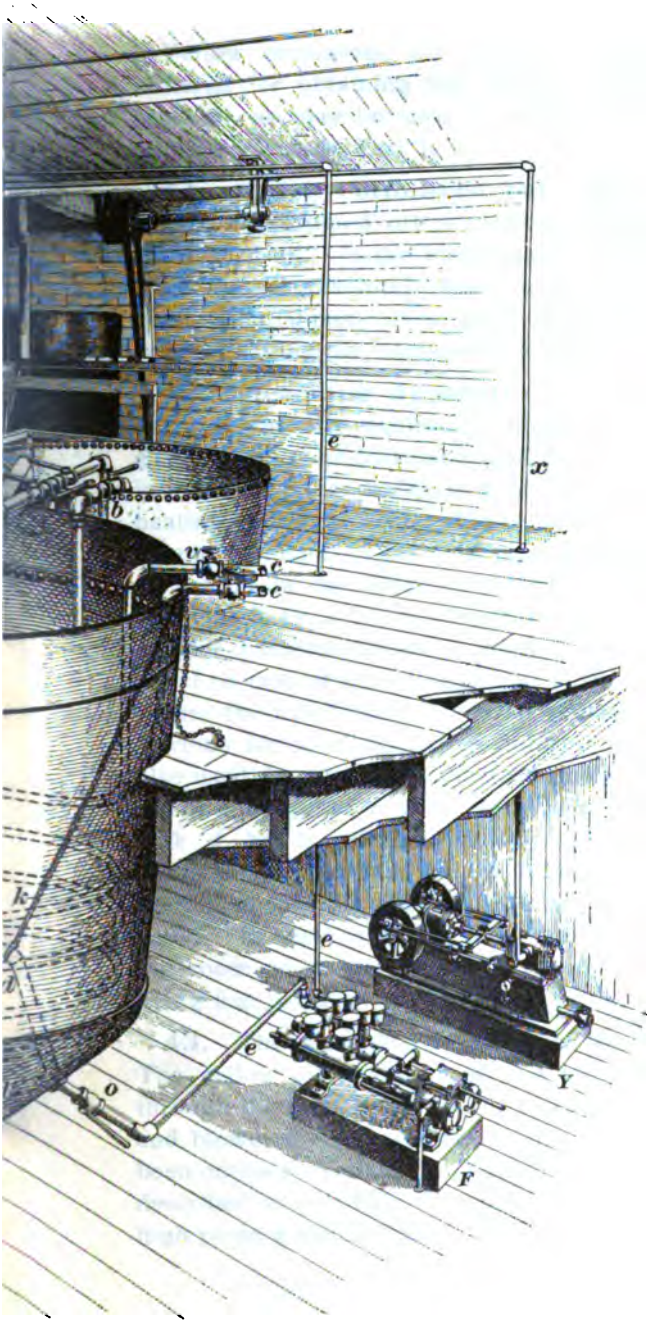


FIG.


e
x
b
c
c
e
e
o
Y
F

Yellow cottonseed oil may be considered as the raw material for obtaining the various other grades of cottonseed oil, as may be seen by referring to Fig. 3, *Cottonseed Oil and Products*, Part 1.

41. White Cottonseed Oil.—The grade known as **white cottonseed oil** is made by bleaching choice yellow cottonseed oil. In the bleaching process, it is necessary to have the oil absolutely free from all moisture. This is a most essential point, as no satisfactory bleach can be obtained without having the cottonseed oil in this condition.

42. Bleaching Apparatus.—In Fig. 2 is shown the apparatus usually required for bleaching by means of fullers' earth. The cone-bottomed tank is shown at *A*. This tank is made of either boiler iron or steel and is provided with heating coils *c*, a valve *v* that regulates the steam, a blower pipe *x*, a siphon, or draw-off pipe, *d* with a swivel joint, and a cock *m* in the apex of the cone. The blower pipe is terminated by a circle of perforated iron pipe of slightly smaller diameter, the perforations having an aggregate area equal to that of the blower pipe *x*, and no more, so as to secure the full benefit of the air pressure from the blower. Various forms of apparatus differing from that shown are also used, but the same general principles apply to all.

The filter press *R* is in close proximity to the bleaching tank *A*, but is protected in any suitable manner from the flying dust occasioned by the fullers' earth being added to the oil, so that the finished, filtered oil will not be contaminated. In most cases, the filter press is placed in an adjoining room so as to afford the desired protection from the flying fine particles of the fullers' earth.

43. The bleaching is done in the following manner: The yellow oil having been run into the tank *A*, Fig. 2, through the pipe *b*, the blower is started and the oil heated and blown, if circumstances require, until the moisture has been entirely eliminated. This is ascertained in the manner described in Art. **17**. The temperature may be carried to a high point within limits, as the hotter the oil, the better the

fullers' earth will effect the bleach. A good temperature for bleaching yellow oil is from 190° to 210° F. The heating and blowing being concurrent, when the oil reaches the temperature just mentioned, all the moisture has been expelled. Enough fullers' earth is now added to accomplish the bleach. The manner of approximately ascertaining the quantity of this material required will be described later. The valve *o* is opened after the oil has been agitated for a few minutes, and the oil and clay are pumped through *e* to the filter press. The latter always has a connection leading from its trough back to the bleaching tank, so that the first portions of the oil may be returned for a more perfect bleaching. The circuit of the oil from the bleaching tank, through the filter press, and back to the tank is maintained until a sample of the oil in the test glass is seen to be of the required white color.

44. The coloring matter having been entirely removed by the bleaching process, the oil is directed through *j*, Fig. 2, by changing the three-way cock *g*, to the storage tank *H*, where it is allowed to remain until it has cooled to the room temperature before being run into barrels. This cooling is necessary in order to obtain the oil in a clear, sparkling condition. If drawn into barrels while it is hot, the oil will be cloudy, and the market value of an otherwise perfect oil will be materially depreciated. During the pumping of the bleaching oil from the tank, the agitation of the material is continued to prevent the fullers' earth from settling to the bottom of the tank. If allowed to settle on the bottom, the fullers' earth would not exert its full bleaching action on the oil.

45. After all the material has been pumped from the bleaching tank, the air from the blower is shut off from the tank and turned on to the filter press to remove the bleached oil held between the plaits and in the cloths. The press is then blown until no more oil escapes from the outlets. If the press is not desired for immediate use, steam is blown through it to steam out all oil held by the clay and the cloths.

This oil should not be mixed with the previously bleached material, but should be saved by itself and treated in subsequent operations. The cloths are then dried by blowing the air from the compressor through the press.

If the filter press is to be used within 24 hours for the same purpose, the steaming operation is not performed, provided the quantity of fullers' earth used in the bleaching has not filled up the space between the filter-press plates. When a large quantity of clay has been used, it is necessary, after the oil has been steamed out and the cloths dried, to remove the dried clay from the latter by scraping them with a dull-edged scraper. The same cloths may then be used without removing and washing; this, however, must in any case be done after the cloths have been used on the filter press three or four times.

46. In bleaching yellow cottonseed oils, it is always advisable to perform a test on a laboratory scale to ascertain their bleaching qualities. It will frequently be found that some yellow oils cannot be bleached to a water-white color and also that in some cases such an excessive amount of fullers' earth is required to obtain a water-white color that it is not profitable to attempt to bleach them. Some yellow cottonseed oils are not susceptible of bleaching to any degree; hence, the advisability of always making the preliminary test on a small scale is apparent. Cottonseed oils are sold in the trade under the name of *bleaching oil*. The quality of the fullers' earth used for bleaching purposes is very important. Its effect in the bleaching of cottonseed oil will be discussed later.

47. In bleaching yellow cottonseed oil in the tank, it is always advisable to use as low a temperature on the oil as will effect the bleach. The lower the temperature at which it is bleached, the better will be the flavor of the oil produced. Certain yellow oils bleach at a much lower temperature than others of the same grade; hence, no rule applicable to all oils can be given. If too high temperatures are used in bleaching yellow oils, they are liable to be burned, and then it

is not possible to bleach them. Too much fullers' earth is always to be avoided in this process, as an excess beyond the quantity required to produce the white oil imparts to the bleached product an objectionable taste and flavor, known as an *earthy* or *clay* taste or odor.

There are two grades of white cottonseed oil, *prime white* and *off;* the difference generally consists in a difference of flavor or of color. Winter-white and summer-white oils are made, the difference between them consisting solely in a difference of results with the cold test, which will be explained later.

48. Preliminary Bleaching Test.—In testing yellow cottonseed oil to determine to what extent it may be possible to bleach it, a 4-ounce sample bottle is filled with a known weight of the oil, 100 grams being a convenient quantity. The bottle and oil are heated on a steam bath to 180° F., a quantity of fullers' earth equal to the percentage judged to be required is added to the hot oil, the bottle corked, and the clay intimately mixed with the oil by violently shaking the bottle for 3 or 4 minutes. The contents of the bottle is then poured into a folded filter, and the filtrate is allowed to run into another bottle. As the first portion of the filtrate is likely to be clouded more or less with fine particles of clay, it is poured back on the filter paper until the filtered and bleached oil passes through perfectly clear. By observing the color and comparing the oil with the original oil for treatment, the capability of being bleached may be judged, and also the approximate quantity of fullers' earth required to produce a white oil. Should the color not be satisfactory with the first test, a second test is made, increasing the percentage of clay added. In this way, a very close approximation may be obtained.

As the air agitation in a tank of oil brings every particle of clay into intimate contact with the material, a far better effect is always obtained in a tank than is possible in the bottle test. For this reason, it will always be found that about one-quarter less clay will be required to effect the

bleach of the oil in the tank than is indicated by the test carried out in the bottle. In the latter, care must be taken to have the oil free from moisture and the bottles perfectly dry before the test is begun.

49. Fullers' Earth.—The material called **fullers' earth** is also known in a technical sense as "clay." It is a peculiar form of clay that, unlike ordinary clay, falls to pieces when placed in water. It has the property of absorbing water to a great extent without becoming pasty. Its most important property is that of absorbing coloring matters from oils. The chemical analysis of fullers' earth gives no clue whatever as to its value as a bleaching agent. Two samples may have practically the same chemical composition and yet differ radically in their physical properties in their effect on an oil subjected to their action for bleaching. While one kind of fullers' earth may give most excellent bleaching results, another with the same chemical composition may have no bleaching effect whatever. It is the concensus of opinion, confirmed by practical experience and investigation, that the bleaching properties of a fullers' earth are due entirely to its physical structure or condition.

50. In the selection of a bleaching clay, care should be exercised to make a test of the sample on a laboratory scale against a sample of known bleaching power for comparison. Equal weights of the clays for the test are taken and the same weight of oil bleached with the fullers' earth and the results noted. Should a new lot of fullers' earth be superior in its bleaching power to that held as standard, the new clay is then retained as a standard for future purchases. There are many inferior bleaching clays on the market, and no large quantities should be purchased without the application of the laboratory test to the sample. The test described for bleaching yellow cottonseed oil may be applied similarly for testing purposes also.

English fullers' earths are extensively used for bleaching cottonseed oils, the color of the earth itself exercising no apparent influence on its bleaching qualities. They are

yellowish and bluish, frequently approaching black in color. Of late years, American fullers' earths have come very much into favor, and some of them do as effective work as the foreign earths.

A fullers' earth for bleaching should be very fine and the clay itself free from sand and foreign material. The finer the clay, the better are the results that can be obtained with it. A bleaching clay should be ground fine enough to pass through a 100-mesh sieve. The drier a fullers' earth is, the better is the bleach that can be obtained with it; hence, it is important to keep the material stored in a dry and, preferably, warm place.

51. Winter Cottonseed Oils.—Most animal and vegetable oils, when chilled, deposit the less readily fusible constituents as stearin. This is especially true of both crude and refined cottonseed oils. On subjecting the settled portion of the oil to filtration and pressure, the more fluid portion, the olein, may be obtained practically free from suspended albuminous matters. The impurities are retained to a great extent in the separated stearin. By allowing cottonseed oil to stand in the tanks at a relatively low temperature for some time after refining, the heavier part of the oil containing the stearin settles to the bottom of the tank. This settling procedure is frequently resorted to as a means of crude separation, as the upper part of the oil, especially in cold weather, may be drawn off and fulfil the requirements of a winter cottonseed oil. The thickened mass remaining in the tanks may then be pressed to obtain the cottonseed stearin, thus effecting a saving of time and much labor in handling.

Oils that remain limpid and clear in a cold temperature (winter weather being in a way understood) are termed **winter oils**, while those that become turbid or partly solidified on chilling are called **summer oils.**

52. Recovery of Cottonseed Stearin in Making Winter Oils.—The making of winter cottonseed oil is concurrent with the production of cottonseed stearin. The

refined oil that is to be resolved into winter oil and cotton-seed stearin is allowed to rest without agitation in a cool temperature—about 30° or 35° F.—until the solid portion of the oil has subsided. This settling, or chilling, is done in receptacles so arranged that the light, thin portion of the oil, the oleins, may be withdrawn without disturbing to any

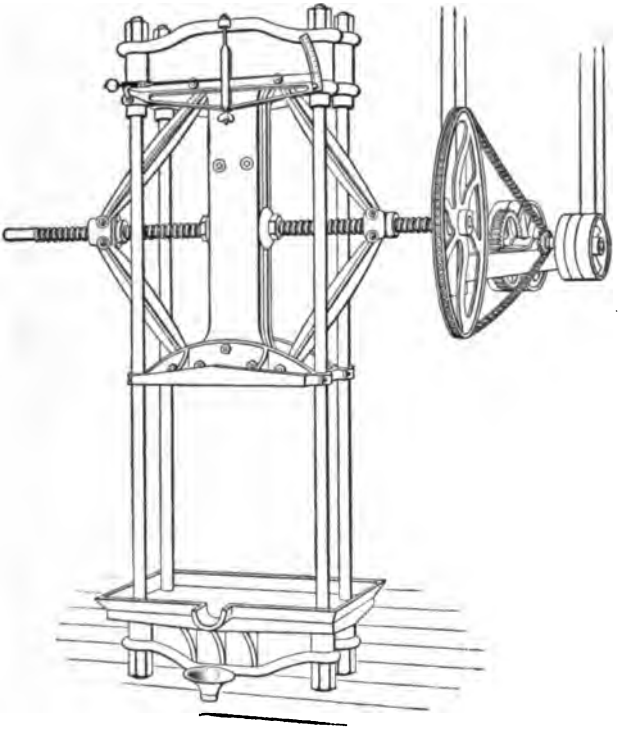


FIG. 3

extent the sediment underlying them. The time required for the separation of the two component parts depends to a great extent on the individual character of the oil under treatment, a period of 10 to 15 days generally being sufficient for the separation; at times, however, the separation is accomplished in a much shorter period. The longer the period of subsidence, the more complete the separation and

the denser the settled stearin portion will be. The tank in which settling takes place may be an ordinary open-head tank in which are placed draw-off cocks at various distances on the upright tank. By means of these cocks the supernatant, clear oil may be withdrawn from the underlying thick portion.

53. The chilled mass, which contains the cottonseed stearin with more or less oil, is removed and then subjected to the usual pressing, the material being encased in duck press cloths of close texture. A press suitable for this purpose is shown in Fig. 3. The thick material is filled into a cloth that is placed over a wooden mold in order that the cakes will be uniform in thickness and of a size adapted to the form. The sides and ends of the cloth are then folded over, thus forming a bag containing the material. Each bag is about 3 inches thick, 14 inches long, and 7 inches wide. When enough bags are ready they are placed in the press in layers, each being separated from the other by thin plates of sheet iron. In placing the bags in the press, care should be taken to place them a sufficient distance apart from one another so as to allow for the spreading of the material in pressing. In this way the crowding together of the pressed cakes will be avoided. A distance of about 3 inches between the bags on the plates will give ample room for the material to spread and press out. The cakes should not be allowed to project beyond the edges of the iron plates, or those portions will be insufficiently pressed. In pressing the molded cakes, the pressure should be very slowly applied, to allow the material to acquire a firm condition gradually. A sudden and quick pressure at any stage of the operation will result in bursting many of the cloths.

54. After the press is filled, the material is first allowed to drain without pressure. When the flow of the oil from the natural weight of the layers one upon another ceases, the pressure is gradually applied by the descending plate, frequent periods of rest being permitted for the oil to drain from the stearin in the cakes. The pressing is continued

until the stearin and the oil have been more or less completely separated, which may be seen by the oil ceasing to drain off under the heavy pressure applied. The press, Fig. 3, is provided, by means of pulleys, with a quick motion for closing up the material and for raising the top plate for unloading, and with a very slow motion, which may be regulated to almost any speed, for the pressing proper.

The room in which the press is placed is cooled by usual refrigeration methods, so that no melting of the stearin takes place. If the stearin were allowed to melt, it would go into the winter oil and cause the latter to show a cloudiness on being chilled for the cold test. The pressed-out oil is conducted to the usual storage tanks and, if necessary, may be run through the filter press to remove any extraneous impurities. This oil is *winter-pressed cottonseed oil.*

55. The solid portion of the cottonseed oil remaining in the cloths is the true cottonseed stearin. It is a light yellowish fat of a buttery consistency and is nearly neutral in its characteristics. It is usually pressed so that it has a melting point of 38° C. There is no standard for the hardness of this stearin, it being sold usually by sample. Within limits, the longer and the harder the mixture of stearin and oil is pressed, the harder the resultant stearin will be; and the lower the temperature in chilling and pressing, the freer from stearin the winter oil will be.

The pressed material is removed from the cloths and disposed of to the trade for various purposes, among them being the manufacture of edible products.

56. Wesson Process Oil.—In the last few years there has appeared on the market a high-grade vegetable oil designated as **Wesson process oil,** one brand being used as a salad oil and another brand as a cooking oil. This cottonseed oil is superior to ordinary cottonseed oils, as it is especially prepared by a secret process and is devoid to a marked degree of the usual characteristic odor and smell. It appears like and possesses many characteristics of a cold-pressed cottonseed oil. The chief claims of the manufac-

turers of this oil are, first, a neutral and palatable flavor and, second, freedom from smoke and odor in frying—points that are not possessed by the ordinary cottonseed oil used for cooking purposes.

57. Oxidized, or Blown, Cottonseed Oil.—The manufacture of **oxidized, or blown, oils** is a comparatively new industry and originated in England. Great hopes have been, and are still, entertained that by the oxidation of cottonseed oil, a substitute for castor oil might be obtained. Up to the present time this has not been accomplished, the use of oxidized, or blown, vegetable oils for lubricating purposes not being as general as was anticipated. Like many new products, extravagant claims have caused it to be undervalued, but it has, nevertheless, a great many meritorious characteristics. The uses of blown oils are increasing, however, and their usefulness is now being generally recognized.

Oxidized cottonseed oil is very similar chemically to castor oil. Its acetyl number (see *Quantitative Analysis*) is about 80, as against 17 before oxidation, and the oxidized product is far more soluble in alcohol (characteristic of castor oil) than the untreated oil. Acids corresponding to sulphoricinoleic have, however, not yet been obtained from the treated product. While this oil may be used, both alone and mixed, for the lubrication of machines of great power, such as hydraulic presses, it has not succeeded in replacing castor oil in soap making, as it will make only dark soaps possessing far different properties from those made of castor oil. Oxidized oils are most suitable and best adapted for making india-rubber substitutes by the action of sulphur or chloride of sulphur. The blown cottonseed oils have many uses that are held as trade secrets, concerning which no data can be obtained.

58. The process of oxidizing is carried out in the following manner: Formerly only winter cottonseed oil, free from stearin, was used for this purpose, but any well-refined cottonseed oil will serve, except for certain purposes. The

oil is placed in a tank provided with means of heating, blowing, and agitation; the bleaching tank *A*, Fig. 2, is suitable for this purpose. The oil is heated without blowing to 212° F.; at this point the air, in finely distributed jets, is sent through the oil from the perforated pipe in the bottom of the tank. The first effect on the oil is to lower the temperature, which soon rises again to a point much higher than the initial temperature. When the temperature has reached 266° F., the blast is shut off and the viscosity of the oil tested. If a sufficiently high viscosity has not been reached through the first blowing, the temperature of the oil is allowed to fall to 212° F. again, when the air is again blown through it until the temperature of 266° F. is attained. The oil is again tested to ascertain its viscosity, which may be compared with a standard sample or with castor oil. If, through the two blowings, the oil has not acquired the desired consistency, the operations of successive blowings and coolings are performed until the required result is obtained.

59. The duration of the operation may be materially shortened by adding to the oil from $\frac{1}{4}$ to $\frac{1}{2}$ per cent. of its weight of a drier. A manganese salt is usually selected for this purpose, the resinate or borate generally being employed. The drier is added to the oil just before starting the air blast, when the oil is at 212° F.

In the operation of blowing and consequent oxidation, care must be exercised not to allow the temperature to rise above 266° F., or the oil will become permanently darkened and thereby greatly deteriorated in sale value. If the high temperature is maintained too long, the oil assumes a tawny, dirty color, and, in addition, such overheated oils are very difficult to mix with mineral oils. Oxidized cottonseed oil resembles closely in appearance ordinary castor oil, but is always more or less sticky.

The oxidizing operation may be performed with oxygen or ozone, but for this purpose the necessary apparatus for generating these gases must be added to the equipment.

COOPERAGE

60. In the cottonseed-oil industry, **cooperage** is one of the most important features tending to profit or loss. The profit in some seasons is so small that losses arising from the use of poor oil barrels may completely obliterate it. For shipping oil in barrels it is a most excellent investment to use only the best, well-seasoned packages.

Barrels made from cross-grained or sappy, green wood should never be used for shipping cottonseed oil. Staves from the former are very easily cracked in handling, thereby allowing a constant leakage in transit or in storage. When green or sappy wood is used, the drying out causes the wood to shrink, thus opening the cracks between the staves and causing such packages to be useless until recoopered, or, as it is technically termed, *trimmed.*

61. Trimming of Barrels.—The operation of removing each head of the barrel and then inserting between each stave pieces of dried flag is called **trimming.** The flag, being of a dry, spongy nature, completely fills the cracks between the staves and accommodates itself to the varying pressure on the iron hoops when driven up as tight as possible. When all the interstices between the staves around the heads are filled with flag, the heads are again put in and the package made as tight as possible by driving the iron hoops until they will stand no further stress. Before filling, the oil barrels should be lined with some impervious material, such as glue or paraffin.

62. Lining of Barrels.—When it is desired to line a barrel with glue, about 2 or 3 quarts of a heavy, hot, glue solution is poured through the bung hole of the barrel that is ready to be filled with oil. When the bung is replaced, the barrel is rolled and turned over in every direction so as to coat every part of the inside surface thoroughly. The barrel is then placed, with the bung out, over a trough and allowed to drain free of the excess of glue solution, which is collected and used over again. By this treatment, all pin

holes (worm holes) are stopped up and any cracked staves coated so that they will not allow oil to leak out. It is customary to line the barrels in the afternoon, so that they may dry overnight and be ready for filling the next morning.

In using paraffin for lining oil barrels, care should be exercised to choose a paraffin of comparatively low melting point. Material of this nature having a melting point of 126° F. is suitable for this purpose. When paraffin of a high melting point, for instance, 136° F., is used for lining, it is, when cold, apt to crack if the barrels are subjected to falls or to rough handling.

Silicate of soda has been used for lining oil barrels, but it has fallen into disfavor, owing to the liability of some of the oil to become partly saponified when placed in barrels in which the silicate of soda has not had time to dry thoroughly hard. Oil placed in such packages becomes milky and very cloudy and consequently much deteriorated in value.

The labor attached to the lining process is much lessened by selecting barrels made of sound and well-seasoned wood. In fact, the best and most economical investment in every way is to use for oil barrels only the best possible cooperage material obtainable.

APPLICATION OF THE FILTER PRESS IN THE COTTONSEED-OIL INDUSTRY

63. The use of the **filter press** in cottonseed-oil milling is of quite recent date. Even at the present time there are many mills that are not equipped with this important piece of apparatus, by means of which large quantities of material may be handled and manipulated rapidly and inexpensively. Its use is objected to by some oil millers for reasons known only to themselves. In every industry where filtration or clarification is a requirement in the various manufacturing processes, the filter press has proved its value beyond all question.

In the clarification of vegetable and animal oils, as a part of the refining process, the filter press is accorded a prominent place. So effectually does it accomplish the work of

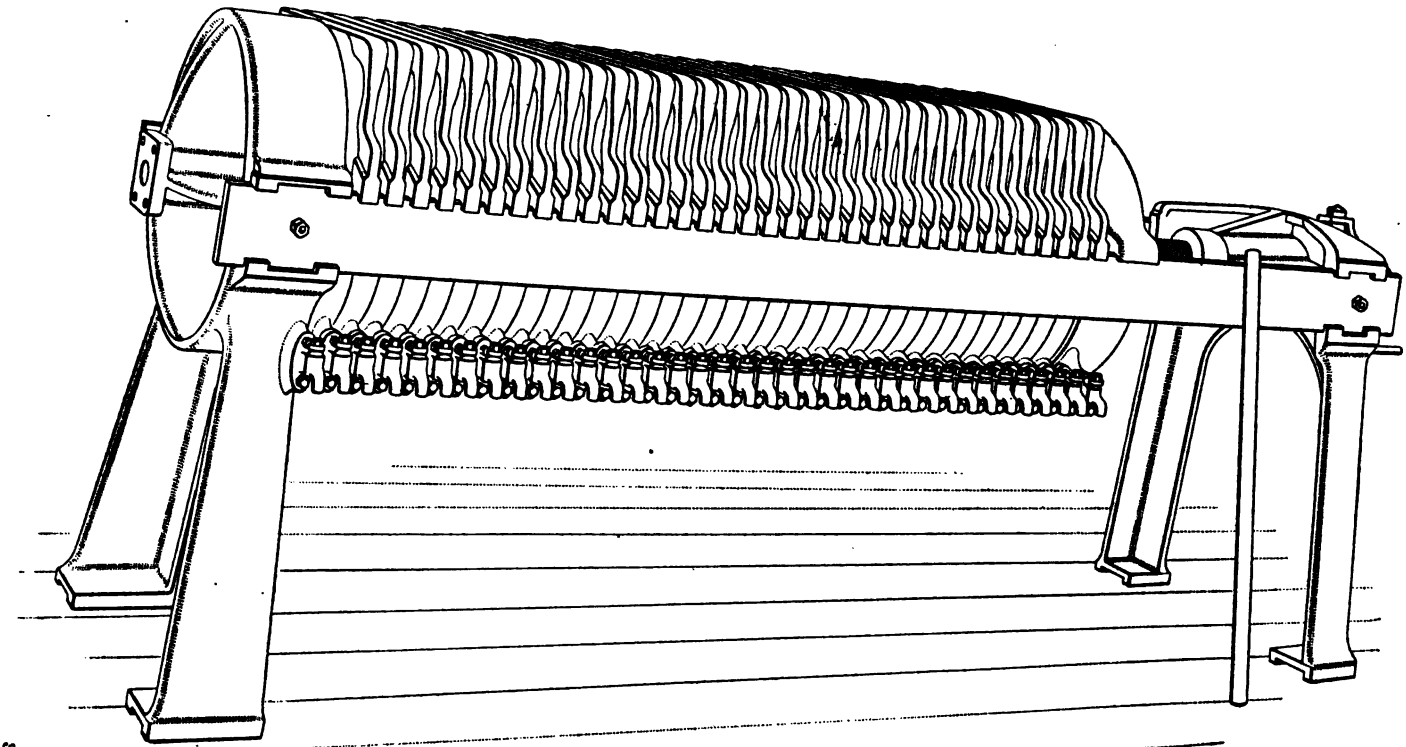


FIG. 1

refining, with especial reference to the elimination of solid and generally extraneous substances, that, in the United States, all former methods for the accomplishment of the same purpose have become obsolete. The requirements of this age for a superior and more quickly prepared finished product have become too urgent to permit of the continued use of the old-fashioned settling tank and gravitation filter.

64. In the cottonseed-oil industry, the filter press is of great economical importance in many ways. By its use it renders practicable the transformation of the crude oil into a merchantable product within a few hours after its extrac-

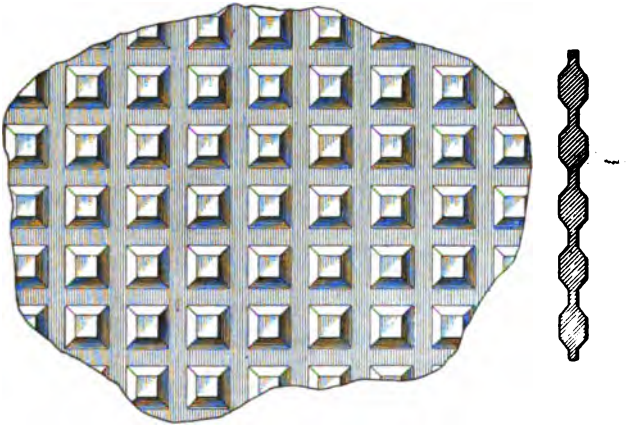


FIG. 5

tion from the crushed cottonseed. Previous to its use in this industry such a proceeding was impossible of attainment, owing to the long period of rest required for the precipitation of the foots and fine vegetable matter in the production of a clarified oil. It is a well-known fact that oil drawn from settling tanks, such as were used previous to the installation of the filter press, in many cases would show some particles of solid and fine extraneous matter even after months of repose. This proved such a serious drawback that the inventive ingenuity of refiners brought forth a number of methods having for their object the separation

of the solid impurities from the oil in which they remained suspended.

While slight improvements were effected from time to time, defective work under the systems that then prevailed proved detrimental to the trade in general. The gummy or mucilaginous substances, the ever-present accompaniments of expressed cottonseed oil, are naturally very slow of subsidence and precipitation. Their complete removal becomes absolutely necessary to obtain a clarified, or refined, cottonseed oil. This removal is effected in a thorough and very rapid manner by means of the modern filter press.

65. Filter Press.—The type of filter press shown in Fig. 4 is one of the most modern, and has lately been installed in several of the largest cottonseed-oil refineries as being especially adapted for this class of work. One of the improvements over the usual style of press is applied to the filter plates, which, instead of simply being grooved, are studded all over the surface with small, flat-topped pyramids. A section of such a plate is shown in Fig. 5. The pyramids support the filter cloth, and at the same time afford an effective passage for the filtrate to pass away, making a marked improvement in the rate of filtration. The equipment, with regard to the filtering medium, is described later; a single plate of this press is shown in Fig. 6.

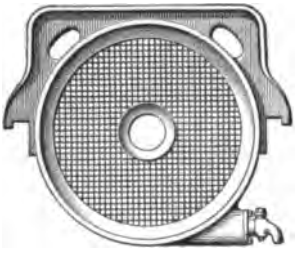


FIG. 6

The cloths having been adjusted on all the press plates, the latter are forced together by a follower, which is actuated by a screw operated by a lever wheel. A very recent device for closing the press is the ratchet lever. This is of great efficiency for the rapid closing and opening of the filter press, and effects a great saving of time and labor. It is constructed on the principle of the ordinary ratchet gear and is operated by the

oscillation of the lever. The plates of the press are recessed, so that a chamber is formed between each two plates. The concave surfaces of these plates are provided with the pyramid surfaces described for the drainage of the filtered oil, which passes through the opening in the bottom of the plate.

66. An improved filter plate has recently been devised. The improvement consists in having a channel running entirely across the lower portion of the plate, as shown in Fig. 7. By means of this channel the liquid is not held

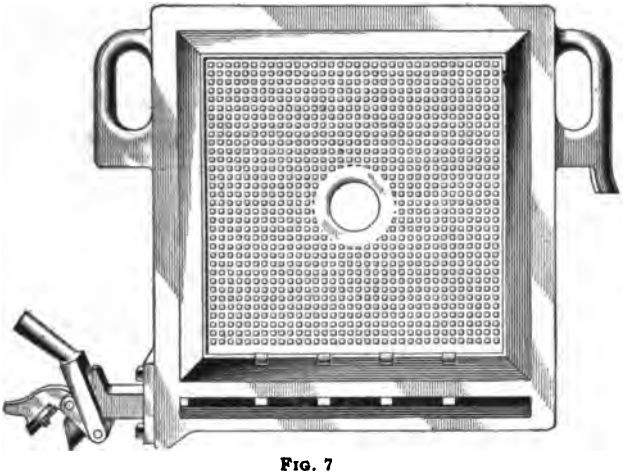


FIG. 7

back, as in the old-style plate, but runs freely through the delivery cocks or through the delivery channel in a closed delivery press. It is claimed that this improvement increases the capacity of the filter press about 25 per cent. These plates are made corrugated or pyramidal and in the round or square form.

67. In effecting the filtration of the oil, the material is pumped into the press, and, passing through the cloths, courses downwards through the interstices in the plates, through the outlet, into the receptacle beneath, a perfectly clarified product, the residue—the mealy and mucilaginous matter in the case of crude cottonseed oil, and the fullers'

earth in the case of bleached oils—being retained in the chambers of the press by the filter cloths. The capacities of filter presses vary with their size, some being capable of handling 40 tons of oil daily.

The construction of the filter press combines the advantages of the largest possible filtering surface in the smallest possible space; the facility of forcing the material through the filtering medium by the most suitable pressure from a slight hydrostatic pressure to a working pressure of 200 pounds per square inch; the ease and facility with which the press can be handled; and the rapid and complete separation of the oil from the residuum and suspended matter. Filter presses are made both of square and round pattern, to suit individual preference, as the shape is wholly a matter of working convenience.

68. It is often desirable to filter a small quantity of oil, such as would fill only part of the filter press. In such a case, the press has inserted at any convenient part a solid plate without the usual center feed-hole and outlet. This plate, known as a *dummy plate*, is shown in Fig. 8.

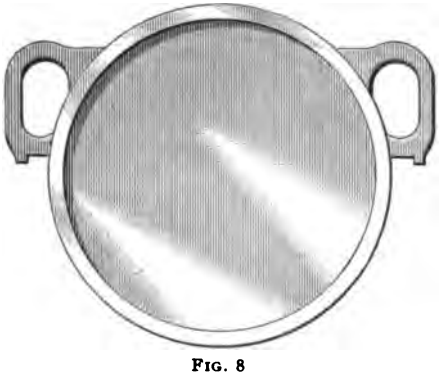


FIG. 8

It is used when it is desired to utilize less than the full capacity of the press, this being accomplished by removing one or two plates and inserting the dummy plate to cut off the flow. For instance, if only five chambers of the press are wanted, the sixth and seventh plates are removed and the dummy plate inserted, making it practically a perfect working press of five chambers. This is found very serviceable in manipulating small, experimental lots of oil. For square filter presses, the dummy plate is naturally made in the square form.

69. Rate of Filtration.—The rapidity with which a liquid is separated from a solid during filtration depends primarily on the force with which it is urged through the filtering medium and the resistance with which the medium opposes the flow. An increase of force in urging filtration results in an increase in the quantity of liquid filtered during a given time. For this reason it is customary to use a pump to force the liquid to be filtered through the filtering medium. An increased flow with an increased pumping pressure is not, however, a universal rule, as in some cases an increase of pressure actually diminishes the rate of filtration. For every substance, there is a certain rate of flow, and hence a corresponding pressure, beyond which there results no increased useful effect to correspond with the increased force applied. A little consideration will show why this is so. Coincident with an increased rate of flow there is an increased resistance at the point of filtration, because the real filtering medium is not the particular fabric or surface on which the solid matters are retained, but the aggregated solid matter itself, which is built up or deposited on this surface. Hence, the greater the volume of flow of filtrate, the more solid matter is deposited, the thicker the filtering bed becomes in a given time, and the closer the particles deposited are driven together under the greater force, and thus, being more firmly compressed, the greater is the resistance offered the passage of the liquid. It is thus apparent that to secure the most favorable permeable condition of the substance deposited there must be a limit, not only to the rate of deposition, but also to the thickness of the deposit, beyond which useful filtration practically ceases.

70. With crude cottonseed oil, with the gummy and somewhat flocculent substances present, the thickness limiting the useful rate of filtration is likely to be soon reached. In this case, any increased pressure over that which is necessary to filter the oil retards the rate of filtration and will, if continued, ultimately so close the pores of the deposit that filtration will cease entirely.

In applying the pressure in filtering oils, the pump should be started very slowly and the speed gradually increased until the maximum pressure for securing the best results from the filter press is obtained. This applies with equal force both to the filtration of crude cottonseed oils and to the bleaching of yellow oils.

71. Filter-Press Cloth.—The filter-press cloth used in the filtering of oils is an important factor in producing good, clear, sparkling oils. No matter how good the filter press itself may be, if it is not supplemented by a good filtering medium the effectiveness of the whole process of filtration is to a great measure nullified. In many of the mills, too little attention is given to this important consideration.

The cloth ordinarily used in filtering cottonseed oils is of closely woven texture, more or less elastic, and very pliable. It closely resembles duck, but is woven differently; it is known as *chain cloth* from the appearance and style of weave. This material answers most requirements in the oil refinery and is of moderate cost.

The best material, however, for the filtration of these oils is the press cloth known technically as *lamb skin*. This material is a closely woven fabric having 450 picks, or threads, to the linear inch. One side of the cloth is smooth, the other having a cotton-flannel appearance. The cloth is placed on the filter-press plate with the woolly side next to the flow of the oil, being protected from being clogged and stopped up by another cloth, a *twill* cloth, placed over it. The twill cloth is more or less like the ordinary press cloth, but is not quite so closely woven. This combination of "lamb and twill" is the ideal filtering medium for cottonseed oils, the reason for its lack of general adoption being its rather high cost.

INDUSTRIAL USES OF COTTONSEED OIL

72. The industrial uses of cottonseed oil are very numerous and diversified. Its use is constantly being extended to new fields, and there seems to be no apparent limit to its wide application, as evidenced by the numerous inventions in which cottonseed oil plays a part. Refined cottonseed oil may be used for almost every purpose to which oil is put except for household illumination. It is not practicable, however, to give here an extended account of every use in the arts and manufactures to which cottonseed oil is applicable or adapted, but some of the most important will be described and others indicated and briefly touched upon. The use of cottonseed oil in the manufacture of edible compounds, such as lard and "cotto" compounds and butterine, will be described elsewhere.

73. Soap Stock.—Cottonseed oil is the purest form of **soap stock** on the market and, as the rules of the various boards of trade, etc. prescribe exactly what its condition and qualifications must be, it is thus always uniform in its characteristics as adapted especially for the soap manufacturer. The oil is made into various grades of soap, from low-grade laundry soaps and washing powders to high-grade toilet soaps.

74. Cleaning and Bleaching.—Cottonseed oil free from mineral oil or wax is used by bleachers of cloth in a somewhat novel manner. If the goods are found to contain some off-color places or are spotted in any way, some cottonseed oil is painted over the spots by means of a brush or a sponge. A second treatment of the goods in the scouring and bleaching will then remove the off-color places and leave the goods an even white. The oil impregnates the fiber of the goods, and thus softens the discoloring matter and renders it easy to remove.

75. Culinary Uses.—Summer-yellow oil is used largely in culinary operations, being especially adapted for certain cooking processes. The use of cottonseed oil for cooking purposes has grown rapidly during the last few years and is

still growing. Winter-yellow oil finds extensive use both under its own name and also as "olive" oil for salad oils.

Cottonseed oil is now very extensively used by bakers as a frying medium in place of lard or cooking compounds formerly used. Those engaged in the fish-frying trade, especially in England, use cottonseed oil almost exclusively for frying.

White cottonseed oil is used to a considerable extent in some confectionary trades.

76. Pharmaceutical Uses.—For pharmaceutical purposes, cottonseed oil finds extensive use as a base for liniments, ointments, and similar preparations in place of olive oil, which a few years ago was used for these purposes. Cottonseed oil is an official preparation of the United States Pharmacopœia.

77. Illuminating Uses.—For miners' lamps, the white cottonseed oil is extensively used. An Ohio law provides that only cottonseed oil, pure lard oil, or their equivalent, must be used in mines in that state. Although cottonseed oil itself cannot be used in lamps with chimneys, by mixing it with kerosene oil in varying proportions, an excellent oil, known to railroads as *signal oil*, is made. A suitable mixture for signal oil is 50 parts of kerosene oil to 50 parts of white cottonseed oil. This mixture is burned in brakemen's lanterns, etc.

Cottonseed oil that is properly refined and free from fatty acids and mucilaginous matters, is fully equal to rape oil for burning in lamps adapted for the latter oil. It has a comparatively large consumption for illuminating purposes where its lower cold test is not a drawback. Practically the only superiority of rape oil as a burning oil lies in its low congealing point.

78. Cottonseed Oil as a Substitute for Linseed Oil. An inventor in New Orleans has claimed that cottonseed oil is capable of being degummed to such an extent that it is practically equal to linseed oil for uses in painting, etc. The degumming process has been kept secret by the dis-

coverer, but should it prove all that is claimed for it, a wider utilization of cottonseed oil will be developed. In addition to rendering the oil siccative, the gum that is extracted is claimed to be valuable for many purposes, among them being painter's or gold-size cements and other adhesive purposes. The degummed oil, when submitted to a further process, is rendered as clear as water. Up to the present time, however, the process has not become a marked commercial success.

Refined cottonseed oil is used to a considerable extent as a semidrying oil in the manufacture of some mixed paints for use where fine work is not desired. It may be used in combination with more or less linseed oil in paints designed for rough work. Cottonseed oil has also a somewhat limited use in the manufacture of varnishes.

79. The following formula gives a substitute for linseed oil: 105 pounds of light Burgundy pitch is melted and mixed with 54½ pounds of raw cottonseed oil and 10 pounds of thick Dutch stand oil, both having been previously heated to 176° F.; 70 pounds of petroleum is now added and the whole heated. When cold ¼ pound of tincture of valerian is added. If the raw cottonseed oil is previously heated with 3 per cent. of litharge, the final product will behave as a varnish, but 3½ ounces of resinate of manganese and half that quantity of turpentine answer better than the litharge.

80. Cottonseed Oil in the Manufacture of Artificial Rubber.—It is said that cottonseed oil has a considerable outlet in the rubber industry. By adding 18 per cent. of crude rubber to this oil, an imitation is formed that it is difficult to detect from genuine rubber. Through the action of chloride of sulphur on cottonseed oil, there is produced a viscous, sticky compound, which is the chief component of "factis," or artificial rubber.

81. During the last 20 years artificial caoutchouc and gutta percha have received considerable attention. Research has been stimulated by the early discovery that isoprene, a

hydrocarbon distilled from caoutchouc and oxidized by acids into an elastic mass having the properties of the natural gum, can be obtained also from gutta percha, turpentine, and vegetable oils. The experiments having shown that the artificial gum can be produced, the problem now is how to lessen the cost. The so-called new synthetic gum of M. Eugene Turpin, who is known as the inventor of melinite, is produced by heating vegetable oil from 257° to 302° F. and oxidizing it by a current of air or by barium or manganese dioxide, adding, as the oil begins to harden, 25 per cent., by weight, of colophonus rosin, 25 per cent. of powdered sulphur, 5 or 10 per cent. of spirits of turpentine, and 1 to 2 per cent. of carbon chloride. On reaching the proper pasty consistency, the mass is cooled and is the artificial caoutchouc. For gutta percha, the formula is varied by omitting the sulphur and spirits of turpentine and greatly increasing the proportion of rosin. The gum is also produced by a wet process, in which the mixture of vegetable oil and rosin is oxidized in boiling water by nitric acid.

82. An artificial product, which for certain purposes can take the place of india rubber and gutta percha, is obtained by mixing oxidizable vegetable oils (linseed oil, cottonseed oil, palm oil, etc.) with tar, creosote, or wood vinegar. Melted or pulverized shellac or shellac solution may, besides, be added. Next, the mixture is treated with diluted nitric acid, producing a non-viscid, elastic, tough product that can be vulcanized.

According to another somewhat modified process, the mass is exposed to the action of nitric acid for a short time only and then heated on plates. The artificial caoutchouc is used either alone or mixed with natural caoutchouc, and is employed chiefly as an insulating material for electric conduits, for waterproofing fabrics, and for insulation on electric wires.

83. Miscellaneous Uses.—Among other uses, cottonseed oil is employed in baths for tempering steel; in combination with other materials for preserving wood by saturation;

for bolt cutting and similar work; for canning fish, and for mixing with higher-priced oils for sophistication, to reduce their cost, the exportation of cottonseed oil to olive-producing countries amply evidencing this fact. A curious circumstance bearing on this question of adulteration has been revealed by statistics, which show that, some years ago, Turkey prohibited by law the admixture of cottonseed oil with olive oil. This action has had a bad effect on the Turkish oil trade, since it has closed most of the purchasing markets against Turkish oils, whose strong flavor can no longer be masked by the aid of cottonseed oil. In some localities, *chip oil* is a trade term for a mixture of olive and cottonseed oil. The composition varies with the individual ideas of the compounder.

COTTONSEED OIL AND PRODUCTS

(PART 3)

MANUFACTURED ARTICLES AND BY-PRODUCTS

MANUFACTURE OF EDIBLE COMPOUNDS

EDIBLE AND LARD COMPOUNDS

1. Lard and edible compounds offer probably the largest outlet for cottonseed oil; they include such edible compounds as cottolene, "cotto" compounds, etc. In former years, owing to the prevailing prejudice against cottonseed oil as an edible material, many subterfuges were resorted to in disposing of these compounds as lards. At present, however, lard and similar compounds are presented to consumers under their legitimate titles of composition, and the advantages of using vegetable oils in place of animal fats have been widely advertised.

2. In 1906, a national pure-food law, known as the Food and Drugs Act, was passed by Congress. This law became operative on January 1, 1907. By the provisions of this law, every article of food in packages is required to bear a label stating its true character, and compound articles of food are required to have stated on the label the various components, or ingredients, of the food product. Hence, the lard compound that was formerly made without the addition or

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admixture of lard, may, under the law, be sold as an edible compound; but the true name of the product must be stated on the label, while the names of the various ingredients, with their various percentages, may be stated if desired. Under the pure-food law, lard compound must have in it an amount of pure lard equal to or greater than any one other ingredient.

3. In the case of compounds containing lard, stearin, or other fats, or cottonseed oil, and in compounds containing stearin and cottonseed oil, the names of the ingredients must appear on the label. If the compound has a distinctive name, as have some of the cotto compounds, the word "compound" need not appear, but the ingredients must be stated on the label. When, also, the word compound is used, it cannot be qualified by any adjective either before or after it, nor can the name of any product be attached to the word compound, unless that product is the principal ingredient of the compound.

The preceding paragraph has been embodied in instructions issued to inspectors and others, and is intended to cover the interpretations of the meat-inspection law regarding trade labels, as well as tentative rulings made by the Pure-Food Commission under the pure-food law.

4. The following description of operations will serve to show how lard compound was formerly manufactured. The product of this manufacture, an edible compound, may be named as choice dictates, but the provisions of the pure-food law must be lived up to.

The proportion of the ingredients used in edible compounds varies according to circumstances, such as the season of the year and the locality for which the compounds are designed. The ordinary and usual proportions for edible compounds are cottonseed oil, 80 per cent., and oleo stearin, 20 per cent. These are the proportions used in winter or for cool climates. In summer or for warm climates, the proportions are cottonseed oil, 75 per cent., and oleo stearin, 25 per cent. These formulas may be, and are, varied by individual manufacturers.

In addition to this, other materials, such as edible tallows, may be added. When oleo stearin is high in price, it is, without detriment to the compound, in part substituted by the less expensive edible tallow.

5. Selection and Treatment of Cottonseed Oil. The cottonseed oil selected for compounding should be as heavy bodied as possible, as the greater the amount of cottonseed stearin it contains the smaller will be the proportion of tallow or oleo stearin required to impart to the compound the desired or required stiffness. Oils that show a deposit of the cotton stearin on standing in a moderately warm place should be given preference over those that remain clear under the same conditions. The oil selected—prime yellow oil—is bleached with fullers' earth in the manner previously described. It is not necessary in bleaching cottonseed oil for the usual edible compound to have the oil water white, as the compound is usually of a more or less creamy tint, and in addition to this, the agitation or chilling that the compound receives previous to drawing it into packages has the effect of making it whiter. The edible tallow and oleo stearin when employed for this purpose are bleached in a manner similar to that followed in bleaching the cottonseed oil. The ingredients are bleached separately and are kept apart until needed for mixing, when they are placed in the mixing tank. Here they are kept mixed by suitable agitation and are pumped through the filter press. They then flow to the agitator or over the chilling roll, commonly termed the *lard roll*.

6. Apparatus Used.—In Fig. 2, *Cottonseed Oil and Products*, Part 2, is shown the general arrangement of the apparatus required for making edible and lard compounds. The tank *A* serves for a mixing as well as for a bleaching tank. The temperature at which the tallow or oleo stearin is bleached should not exceed 120° or 130° F.; otherwise, a strong and tallowy taste is likely to be imparted to the material. The same general directions previously given relative to bleaching apply to the bleaching of these materials.

A most important point in bleaching any oil or fat is to have it "dry," or perfectly free from moisture. If such is not the case, the fullers' earth when added to such material will at once absorb the moisture present and become pasty. When in this condition, the fullers' earth soon prevents the cloths on the filter press from allowing the bleached material to pass through them. This practically necessitates starting the operation again under the right conditions, thus wasting both time and material. In addition to this, oil or fat treated under these unfavorable conditions is more than likely to acquire an undesirable and objectionable earthy, or clayey, taste.

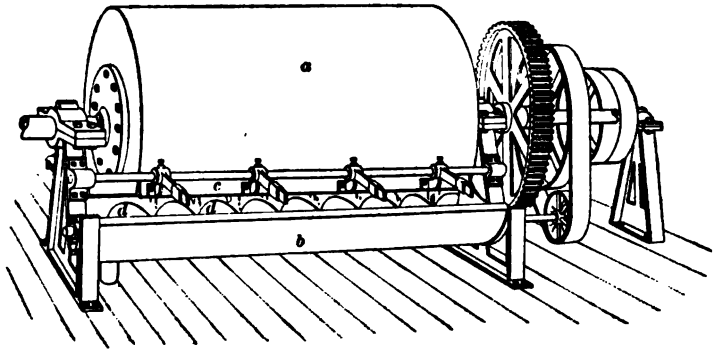


FIG. 1

7. The **agitator** used for chilling edible and lard compounds is of the usual style of lard agitators. A common form is an open, cylindrical tank of boiler iron, containing a revolving shaft to which radial arms are attached. The tank used for the agitator has a double shell through which cold water or brine circulates and thus assists in rapidly chilling the warm compound. The material in the agitator is stirred by the revolving arms until it is almost solid; that is, as stiff as it can be and yet in a condition to be drawn or pulled from the agitator by the pump for filling the various packages. The compound when put into an ordinary lard pail should be of such consistency that when the pail is held upside down immediately after filling, the contained material will remain in it.

8. Lard Roll.—If large quantities of edible or lard compound are made daily, the method just outlined seldom fulfils the requirements of rapid cooling. In such cases, the **lard roll,** the general form of which is shown in Fig. 1, is used. The iron roll *a* is cast in one piece and varies in length from 4 to 8 feet, being usually 3 feet in diameter. In some cases, two rolls are used in connection with each other. One roll, which is chilled by ice water, receives the hot material, and the other, which is chilled by brine, finishes the chilling. The hot material is fed from a tank above the roll, or rolls, at a rate that allows the compound to be perfectly chilled in one revolution of the roll, the speed of which is about 10 revolutions per minute. When the chilled compound just about reaches the attached trough *b*, it is removed from the roll by an attached scraper *c* that causes the material to drop into the trough. From the trough the material is removed by a revolving screw *d* to an adjacent agitator, where the lumps formed on the cylinder are broken up. From the agitator, the material is drawn into the packages. Frequently, the compound is removed from the trough by a pump that draws the material through a screen in the end of the trough, thus breaking the lumps and rendering the stock suitable for immediate drawing into packages. The lard roll is always in connection with the refrigerating system of the plant, to insure a constant cold temperature.

A recent improvement on the lard roll provides for feeding the mixed compound on the side of the roll and permits the compound to be in contact with the refrigerated surface for a longer time before being removed by the scraper.

9. The main object to be achieved in making edible and lard compounds is to have them contain as large a percentage of cottonseed oil as possible and yet fulfil the required conditions as to the stiffness of the material to withstand warm temperatures without much softening. In recent years, articles known as cotto compounds, such as cottolene, cotto suet, etc., were put on the market to compete

with lard compound as formerly made. These compounds are practically lard compounds (as filling the old-style definition), being made with the yellow, unbleached cottonseed oil instead of with the white oil. The proportions and methods of making are identical with those that obtain with the usual compounds, frequently having the oleo stearin substituted by beef fat or suet. In the case of cotto compounds in general, the process of bleaching the various materials is not resorted to, the materials being simply mixed and filtered in the manner described, and labeled as the pure-food law requires.

10. Lard Compound.—As manufactured for use in the United States, **lard compound** must contain a certain proportion of pure lard. The processes described in the preceding paragraphs apply in every detail to the manufacture of lard compound when made with more or less pure lard. The quantity of the latter to be added to the edible compound is a matter entirely controlled by the individual manufacturer, but he must comply with the sections of the pure-food law covering this point.

For some export trades, a certain quantity of lard was formerly mixed with the old-style lard compound, which mixture, as then made, would now fulfil the conditions required by the national pure-food law.

Another requirement of this law is that "in all cases, only sweet and clean edible stearin and sweet and clean edible fats shall be used" in the making of lard compounds, compounds, or lard substitutes.

BUTTERINE, OR OLEOMARGARINE

11. Formerly, one great outlet for cottonseed oil was its use in the manufacture of **oleomargarine** or, as it is commonly known, **butterine.** The manufacture of this article has greatly declined within the past few years, owing to the enactment of a federal statute that went into effect on July 1, 1902. This law imposes a tax of 10 cents per pound

on oleomargarine used for domestic consumption and in which yellow coloring matter has been introduced. At present, in consequence of this practically prohibitive tax, there are only a few factories making this product. Many of the factories that were formerly engaged in this industry have turned their attention to the renovating of butter and the blending and reworking of the varied assortments of country butter into large lots of uniform color and character. This comparatively new industry has made great progress, still it is not an adjunct of any cottonseed-oil mill or refinery.

12. The quality of cottonseed oil used for the manufacture of oleomargarine is that known as butter oil. Oleomargarine was invented in 1870 by Mége Mouries, a celebrated French chemist, on an order from the French government to provide for the army a cheap and wholesome substitute for butter. His discoveries and theories in this line have been accepted and confirmed by the food experts of the world as eminently satisfactory and of great value. He demonstrated by many careful experiments that the formation of butter contained in milk was due to the absorption of fat contained in the animal tissues and that the oil pressed from the sweet fat of beeves corresponded exactly with the oil found in the natural butter. The oil expressed from beef fat is known as *oleo oil* and possesses a bland taste and faint buttery odor. Its color is usually similar to that of butter. While the original name, oleomargarine, was applied only to the product oleo oil, it is now applied to the modern mixture of oleo oil, cottonseed oil, neutral lard, cream or milk, with the usual salt and with or without coloring matter, known as butterine. As indicated, the terms oleomargarine and butterine are used synonymously in the United States but not in European countries.

The ingredients constituting butterine vary in proportion with the individual ideas of the manufacturers and according to the season of the year or the climate to which it is to be shipped.

13. Manufacture of Oleo Oil and Neutral Lard. The products known as **oleo oil** and **neutral lard** are frequently manufactured in connection with the making of butterine.

Oleo oil is made as follows: The choicest parts of the beef fat—the caul, ruffle, and heart fat—are chilled and thoroughly washed immediately on removal from the slaughtered bullock. The fat is chilled for 10 or 12 hours in water, the temperature of which is slightly above freezing, and then removed from the water and allowed to drain for about 1 hour. The material is then cut, or "hashed," very fine, falling into a water-jacketed kettle provided with mechanical stirring paddles, where it is melted at a temperature not exceeding 170° F. for about 2 hours. The water and scrap settle to the bottom of the kettle, the clear oil on top being siphoned off to another kettle, where it is allowed to settle for 3 or 4 hours, or for even a shorter period. The warm material is then drawn off into receptacles and kept at a temperature of about 90° F. to "grain"; that is, to cause the separation of the solid part of the fat—the stearin—from the liquid portion—the olein—the latter constituting principally the oleo oil. The grained material after "seeding" for 24 hours is placed in cloths of heavy, closely woven duck and subjected to pressing by a powerful press, similar to that shown in Fig. 3, *Cottonseed Oil and Products*, Part 2. The solid portion of the fat remains in the cloths and is known as oleo stearin. The oily part expressed is oleo oil, which after a short period of settling is ready to be used for mixing with the other ingredients.

14. Neutral lard is made from the leaf lard of the hog. After chilling in a cold atmosphere for 24 hours, the leaf lard is hashed and cooked in the same way as the fat for oleo oil and is separated in the same manner after cooking. The liquid neutral lard is allowed to settle for about 4 hours, so as to become free from impurities, when it is suitable for use in butterine. The temperature used for cooking this material does not exceed 135° or 140° F.

Both oleo oil and neutral lard may be purchased from the large packing houses and prepared for use simply by melting.

15. The proportions of the different ingredients used in butterine vary according to circumstances. A large manufacturer of oleomargarine uses the following formula: Neutral lard, 35 per cent.; oleo, 35 per cent.; cottonseed oil, 15 per cent.; cream or milk, sufficient to produce 15 per cent. of butter. This may be taken as a fairly representative mixture. The highest grades of butterine have from 25 to 33 per cent. of creamery butter in them. Some manufacturers claim to produce butterine without the use of cottonseed oil, substituting for it sesame oil, which answers the same purposes.

16. Manufacture of Oleomargarine.—The process of manufacturing oleomargarine is carried out on almost identical lines in all factories. The details of the methods are as follows:

The cream, or in most cases milk, is received in felt-covered cans, to guard against fluctuating temperatures. It is at once emptied into a tin-lined vat and when thoroughly mixed is pumped at a temperature of 80° F. into a centrifugal separating machine, which separates the cream from the milk. The latter is now known as skim milk and is of no further use to the butterine maker. The cream is conducted through block-tin troughs to the sterilizer, where it is pasteurized at 170° F. for a few hours to render inert any germs that may have been in the milk. The cream is then removed and ripened in vats or other receptacles. The period for ripening is from 20 to 30 hours, the cream being kept at a temperature of 70° F. In warm weather, somewhat less time is required for ripening, or "souring," the cream—the condition necessary for successful churning.

The oleo oil, neutral lard, and cottonseed oil are kept in separate storage tanks, which are generally located above the churn. The desired weight of oleo oil is run into the churn at a temperature of 130° F.; the neutral lard is then

added at the same temperature, while the cottonseed oil is added cold, or at room temperature. The ripened cream is taken from the vats at its temperature, 70° F., and added to the other materials in their respective proportions. The object of adding the ingredients in the churn at these different temperatures is to have the mixture at a temperature of 90° to 100° F. at the time of churning. Some makers prefer a lower temperature, about 80° F., for the churning. A temperature that is too high renders the churning process very difficult, as the butter or oleomargarine will not blend, but will remain separated and granular. In any event, the mixture must be churned under 122° F.

17. The **churn** used in the manufacture of oleomargarine is an upright, galvanized-iron cylinder with an open top and having its edges rounded over. This churn is about 3½ feet high and 2 feet in diameter and holds a charge of about 600 pounds. It is provided with paddles attached to a revolving shaft, which is actuated by pulleys and shafting and revolves in a foot at the bottom of the churn.

18. The desired proportions of the different ingredients having been put into the churn, the required amount of *butter color* to produce the depth of yellow color desired is added, the paddles started revolving at a very high rate of speed, and the churning continued for from 7 to 10 minutes, when the operation is finished and the butterine formed. If uncolored product is to be made, of course the coloring matter is not added to the mixture.

The whole mass is then discharged from the bottom of the churn into vats of water. This water is kept at about 33° to 35° F. by means of cakes of ice placed in the bottom of the vats. The butterine is allowed to remain here until it sets hard, which requires about 10 minutes, being moved about occasionally by wooden shovels. The chilled mass is then thrown on inclined tables with the wooden shovels and left there to drain for about 10 hours. The usual custom is to leave the material in this condition over night, so that it may be worked the next morning; for this reason most factories

do their churning in the afternoon. While on the draining tables the stock is salted with the usual amount of fine salt, about 8 to 10 per cent. of salt ordinarily being used. A peculiar feature in regard to salting is the unanimous verdict of oleomargarine makers that Ashton salt, an English production, is the best salt for the purpose. This kind of salt is universally used for salting butterine. During the period of draining, the material is turned over several times so as to eliminate as much extraneous water as possible. The temperature of the draining is kept as near 70° F. as practicable.

19. The butterine is next taken to the butter worker—a machine for mixing and amalgamating the product to a homogeneous texture. This machine evenly distributes the salt throughout the material and at the same time works out the surplus water. The usual process of making up the packages then follows. These packages vary in size from 1 pound to the usual butter tub, or firkin. For export trade, the butterine is placed in various sized cans and hermetically sealed. The packages are kept in cold storage until needed for shipment.

20. Every oleomargarine factory is under strict government supervision, and every package bears a penalty label similar to that required in other industries subject to internal-revenue regulations. At present, a tax of $\frac{1}{4}$ cent per pound is placed on all *uncolored* oleomargarine produced. A daily record is kept of all materials used and of all goods produced, and a monthly report is made, under affidavit by the manufacturers, to the United States Internal Revenue Department.

21. The **butter color,** or coloring dyes, that may be used under the national pure-food law must be harmless. The regulations provide that the names of harmless dyes that may be used in food products be communicated to the inspectors in charge, and that no food product that contains a dye whose use has not been approved by the authorities shall be manufactured for interstate or foreign commerce. At present, only *pure* annatto (see *Organic Chemistry*) is permitted to be used in food products as a coloring matter.

The proportion of coloring matter used for making yellow oleomargarine is determined by individual preference and the usual trade requirements of different manufacturers.

22. No high-grade butterine can be produced without cleanliness, which is one of the requisites of prime importance in its manufacture. The utensils and all parts of the factory are daily cleaned with hot water, and everything used in the whole process is always kept scrupulously clean. In this respect, the oleomargarine factory rivals and almost always surpasses the very best creameries in the country.

23. Bacterial Cultures in Oleomargarine.—Closely associated with the application of bacteriology to butter making is its application to the preparation of artificial butter and various oleomargarine products. In certain European countries, especially in Holland, oleomargarine is made in very large quantities. The largest factories in the world are located in Rotterdam. In these factories, the use of pure bacterial cultures has for some time been adopted with almost absolute unanimity. The shrewd business men that manage these factories have thoroughly learned that if they wish to obtain in their products a flavor imitating that of butter, they are obliged to use bacteria to give them this flavor. They therefore buy the artificial pure cultures and with them inoculate large quantities of pasteurized milk in essentially the same way that the butter maker inoculates his cream. They allow this milk to stand in a warm place for such a length of time as will produce the proper amount of souring, and then this ripened milk is mixed with the fats and the mixed oils and made into oleomargarine products. The result is that a flavor of butter, derived from bacteriological products of the souring milk, is imparted to the oleomargarine. All the better grades of artificial butter are made in this way. Makers of oleomargarine use various kinds of pure bacterial cultures and experiment on them with a great deal more care and know much more about their use than do the butter makers.

Butter makers make comparatively small quantities of butter, while the product of oleomargarine factories is made in very large quantities, and the market for it is much more dependent on the grade of product than is the market for butter. Some of these oleomargarine factories are equipped with bacteriological laboratories, where experiments are going on constantly and where pure cultures are made and advantage is taken of the most recent advances in bacteriology.

COTTONSEED FEEDING STUFFS

24. Cottonseed feeding stuffs include, ordinarily, cottonseed, cottonseed cake or meal, cottonseed hulls, and mixtures of meal and ground hulls. Cottonseed meal has met with great favor abroad for many years, but only of late years has it been consumed to any extent in the United States. It is the most concentrated, nutritious, and one of the cheapest of ordinary feeds for cattle and sheep. In the Northern States, wheat bran is the bountiful supply of feed containing much protein, while cottonseed meal takes its place in the South.

The value of cottonseed meal as a feed stuff is now widely known, and its consumption for this purpose is constantly increasing. It is frequently employed for mixing with feeds low in protein but high in carbohydrates, such as mixtures of corn, oats, and similar cereals. Its use in mixed feeds, when a high percentage of protein is desired, is very extensive. Wheat bran contains about 15 per cent. of protein, while cottonseed meal contains about 43 per cent., or nearly three times as much as does bran. It is thus seen that cottonseed meal affords an abundant and cheap supply of the protein feed. It is highly esteemed as a feed for all animals except hogs.

Many experiments have shown that cottonseed meal produces, pound for pound, more beef than cottonseed itself, but only a limited number of these experiments are of such nature as to give anything like a definite idea of the relative

beef-producing powers of the two materials. An average of three experiments made showed that 1 pound of cottonseed meal has made as much beef as 1.47 pounds of whole cottonseed.

25. Cottonseed Meal.—Numerous experiments have shown that **cottonseed meal** produces in the same rations more and cheaper beef than the same amount of corn meal, and generally more than wheat bran, linseed meal, and the other concentrated feeds. There is abundant evidence to prove that cottonseed meal stands at the head of concentrated feeds. Table I shows the comparative composition of average cottonseed meal and bran.

TABLE I

COMPOSITION OF COTTONSEED MEAL AND BRAN

1 Ton	Water Pounds	Ash Pounds	Indigestible Protein Pounds	Carbohydrates Pounds	Fat or Ether Extract Pounds	Digestible Protein Pounds
Cottonseed meal	164	144	106	584	262	740
Bran	238	116	58	1,258	80	250

One ton of cottonseed furnishes about 750 pounds of cottonseed meal when made without an admixture of ground hulls. An analysis of cottonseed meal for feeding purposes gave moisture, 8.72 per cent.; oil, 12.91 per cent., protein, 43.46 per cent.; carbohydrates, 18.59 per cent.; fiber, 9.72 per cent.; and ash, 6.60 per cent. The composition of cottonseed meal naturally depends on the composition of the seed, the completeness of the separation of the hulls and kernels, and the expression of the oil from the latter, all of which greatly influence the result obtained by analysis. When ground hulls are mixed with the meal or cake, the analysis will naturally show a lower percentage of protein.

26. Cottonseed Hulls.—The **cottonseed hulls,** constituting about 45 per cent. of the weight of the ginned seed, are composed principally of woody fiber and nitrogen-free

extract, which, with water, constitute more than 90 per cent. of the entire hulls. The hulls are sometimes ground very fine and bolted, resembling in this condition linseed meal. This finely ground material is at times mixed in varying proportions with cottonseed meal, such as 1 part of meal to 4 parts of the hulls, the mixture being sold for a combination food as cottonseed-meal bran. To make a product resembling English meal as ground from undecorticated seed, which practically is 55 per cent. of meal and 45 per cent. of hulls, the ground hulls in former years were mixed in the proportions just mentioned with the meal and sold as an English product.

27. The ground hulls, as previously noted, are now merchantable and are sold either alone as *hull bran* or, when mixed with cottonseed meal, as a regular feeding meal, usually with a guaranteed analysis of the manufacturer.

The large amount of fiber on the hulls has been claimed to be no detriment as a feed, as it does not, like cattle hair, produce balls in the stomachs of the animals. But the less lint on the hulls, the better is the product as a stock feed. Cottonseed hulls from different mills, and even from the same mill, vary widely in composition, depending on the thoroughness of separation from the meats, larger or smaller quantities of the finely broken kernels almost always remaining with the hulls. An analysis of the hulls themselves, free from fiber and meats, gives as an average analysis: moisture, 11.36 per cent.; ash, 2.73 per cent.; protein, 4.18 per cent.; fiber, 45.32 per cent.; nitrogen-free extract, 34.19 per cent.; and fat, 2.22 per cent.

28. Hull Bran.—The **hull bran** is obtained by grinding the cottonseed hulls as they come from the huller free from meats. Any suitable form of machine that will grind hulls is suitable for this purpose, the usual kind employed being the ordinary attrition mill.

The grinding device consists of two disks that are mounted on separate shafts and driven in opposite directions at high speed. To these disks are fastened the special hard-metal

segments, or grinding plates. These grinding plates never require sharpening, and when necessary they can be replaced at slight cost, retaining the balance of the disks, a very necessary essential in operating the machine. The material is ground by a compound action—attrition and concussion.

29. In Fig. 2 is shown the form of mill generally used for grinding cottonseed hulls. The material is fed into the hopper *A* and is ground by revolving disks located inside the casing *B*. The pulley *C* that drives one of the disks is run with a straight belt, while the other pulley *D*, which

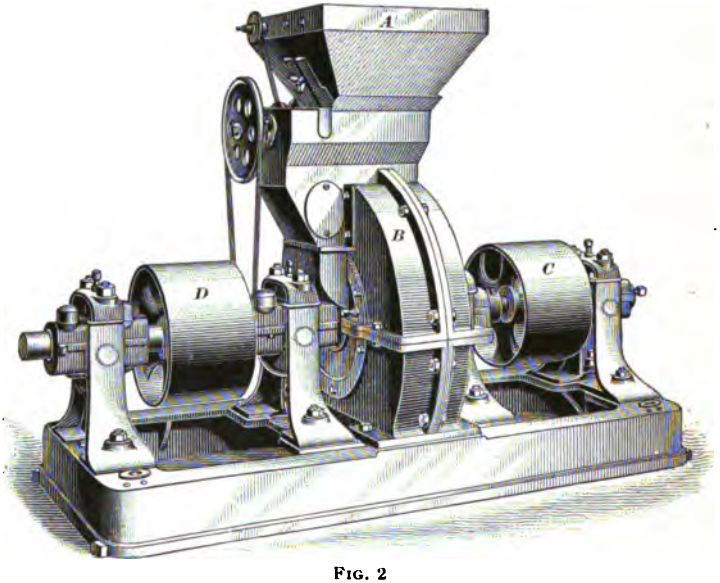


FIG. 2

drives the other disk in the opposite direction, is run by means of a crossed belt. The grinding disks, which are made up of various sections, are shown in Fig. 3. The ground material falls on a screen underneath the grinding apparatus, and is there separated into ground fiber and hull bran. The fiber usually falls over the end of the screen, while the sifted bran is conveyed to any desired part of the mill by means of the usual conveyer. These products are usually disposed of through the regular trade channels.

To be ground successfully, hulls should be as dry as possible; and to obtain the best results in hull bran, it is usually necessary to put the material through the grinder two or more times. The ground fiber when bleached makes good paper stock and, besides, has several other industrial uses.

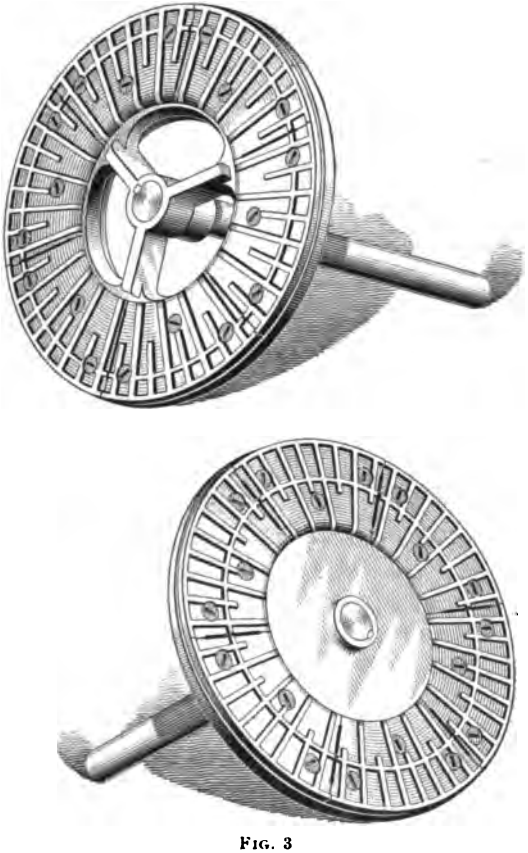


FIG. 3

30. An English patent was recently granted for a process of converting woolly cottonseed hulls into a meal suitable for use as cattle food, etc. The method of preparation is as follows: Cottonseed hulls or other material containing fiber difficult of digestion are thoroughly mixed with about 5 per cent. of their weight of hydrochloric acid of a specific

gravity of 1.16 and heated in a closed vessel, provided with a stirrer, to a temperature of from 212° to 300° F. The amount of acid to be added depends on the material employed and on the duration of the heating. If heating is kept up for 30 minutes, the percentage of acid just mentioned will be required, but the quantity may be reduced if the heating is prolonged. After heating, the substance is ground, and at the same time it is mixed with some basic substances, such as sodium carbonate, chalk, cottonseed kernel meal, etc., to neutralize the acid. During the heating, the acid vapors coming from the mixture may be led into a second quantity of material contained in a separate vessel, air being drawn through both vessels to facilitate the removal of the acid vapors.

This process makes a meal in which the fiber is not recognizable as ordinary fiber, the treatment effectually carbonizing all the lint and hulls to a very dark brownish powder. The analysis of this meal naturally reveals a large percentage of indigestible fiber.

31. Raw Cottonseed.—In the United States, **raw cottonseed** has practically never been fed to animals on a large scale. The lint and the dust on the material are apt to prove injurious, and when fed in excess, cottonseed proves deleterious to the health of the animals. Investigation has also proved that it is a very wasteful procedure to feed raw cottonseed to animals, and knowledge of this fact naturally led to its complete abandonment. An average of twenty-five analyses of whole cottonseed gave the following result: Moisture, 9.92 per cent.; ash, 4.74 per cent.; protein, 19.38 per cent.; fiber, 22.57 per cent.; nitrogen-free extract, 23.94 per cent.; and fat, 19.45 per cent.

32. Composition of Cattle Feeds.—All cattle feeds, whether concentrated, like cottonseed meal, or coarse, like hay, are composed of six groups of substances that the chemist must determine to obtain their value as feed. The term "concentrated feed," taken in its broadest sense, includes the grains and other seeds of agricultural plants, as

well as their many by-products produced in the process of oil extraction and in the preparation of human foods. The groups of substances composing these feeds are *water* or *moisture*, *ash*, *cellulose* or *fiber*, *fat*, *protein*, and *non-nitrogenous* or *nitrogen-free extract*.

Crude ash represents the mineral ingredients of the seed and remains as ashes when the material is burned. This ash consists of lime, potash, soda, magnesia, iron, phosphoric acid, and sulphuric acid, and at times other substances, as chlorine, etc.

Crude fiber, or **cellulose,** is the coarse, or woody, part of the plant or seed. While, as a rule, it is present only to a limited extent in grains and their by-products, it is largely present in cottonseed hulls.

Crude fat includes not only the various fats and oils found in different feed stuffs, but also waxes, rosins, and coloring matters. It is sometimes termed ether extract, because it represents that portion of the material soluble in ether. The fat found in grains and seed is comparatively free from these waxes and rosins.

Crude protein is the general name for all of the nitrogenous matters of the seed. It corresponds with the lean meat in the animal. It has the same elementary composition as animal flesh and is considered the most valuable part of concentrated feeds.

Non-nitrogenous, or **nitrogen-free extract** consists of sugars, starch, gums, and the like. The grains are much richer in starch and similar substances than are the cottonseed products.

33. The fiber and extract matter have the same functions in the process of nutrition, and collectively they are termed *carbohydrates* (see *Organic Chemistry*). The most common and important carbohydrates are sugar and starch. The general methods for the estimation of these substances will be treated later.

Some states have statutes requiring that concentrated feed stuffs be sold with a guaranteed analysis as to the percentage

of protein and fat they contain. A large manufacturing concern sells its prime cottonseed meal as a feed stuff with the guaranteed analysis of protein not less than 44 per cent. and of crude oil and fat not less than 9 or 10 per cent.

Cottonseed meal for domestic trade is always put up in 100-pound bags; that for export, either in 100-pound bags or of weight designated by the buyer.

34. Cottonseed as a Feed for Hogs.—Although cottonseed is used with the greatest success and economy in feeding cattle and sheep, it cannot, as previously mentioned, be used as a ration for hogs without deleterious and nearly always fatal results. It has been used in almost every form for feeding hogs, but never with success when the feeding has been continued any length of time. It has been fed raw, roasted, steamed, and boiled, and the meal, both raw and cooked, has also been tried in various ways, but almost always with the same result—the death of from one-fourth to one-half the drove within 12 weeks from the time the feeding was begun. Apparently the hogs do well the first few weeks after the feeding begins, but at about the sixth week occasional deaths occur, and the losses continue from that time onwards. The hogs show no indication of sickness and make very satisfactory gains until within 2 or 3 days, or sometimes within a few hours, of death, and no curative treatment seems to have any effect. Nearly every experiment station in the Southern States has endeavored to find some combination of feeds, of which cottonseed should be a part, that could be fed with safety, but without success. Before it had any commercial value, cottonseed was often thrown into shallow ponds, where it soon became half rotted, and when in that condition hogs often ate it with impunity. With the present market values of the seed, more than double the amount of food can be secured by exchanging it for corn or similar feeds.

Various experiments have been conducted to ascertain what part of the seed—the lint, the hull, or the meal—was injurious to hogs and the manner in which it acted. Recent

French investigations appear to show that the injurious effect of cottonseed is due to a poisonous principle peculiar to the kernel.

Investigations as to the feeding of hogs on cottonseed meal are constantly being made in the United States, but as yet no entirely satisfactory results have been obtained, although some feeders claim that they have succeeded in obtaining good results with cottonseed feed. At present, whether it is wise to feed cottonseed to hogs is a debatable question, with the preponderance of opinion against the practice.

COTTONSEED-FERTILIZER MATERIALS

35. The principal fertilizing elements in cottonseed products are the well-known combinations of the elements nitrogen, phosphorus, and potassium, which have already been discussed (see *Quantitative Analysis*). These elements are furnished by the cottonseed, cottonseed meal, and the hulls. The ashes from the latter furnish a large proportion of potash and also phosphoric acid.

36. Whole Cottonseed.—In former years, when cottonseed was practically of no commercial value, the whole cottonseed was used extensively in a wasteful way as a fertilizer in the South, care being taken to kill the seed first, by fermentation or by rolling, so as to prevent germination. At present, the practice of fertilizing by whole cottonseed has been practically abandoned because of the enhanced value of the seed and the fact that better fertilizer can be purchased in the form of cottonseed meal and mixed fertilizers. Many cottonseed mills are accustomed to offer to exchange with the producers of the seed a stipulated weight of cottonseed meal for each ton of cottonseed, the quantity of meal offered varying with the ruling prices of seed. This exchange is always advantageous to those using the seed only as fertilizer.

37. The fertilizing constituents of cottonseed are indicated by the following analysis, which is the average of

fifteen analyses of different samples of seed: Moisture, 8.42 per cent.; ash, 3.78 per cent.; nitrogen, 3.13 per cent.; phosphoric acid, 1.27 per cent.; and potash, 1.17 per cent. When raw cottonseed is used as fertilizer, the full value of the constituents is not obtained as in the case of meal, as the physical condition of a fertilizer exercises a great influence on its availability. For fertilizing purposes, a ton of seed is about equal in value to 800 pounds of cottonseed meal, the superior physical condition of the latter being taken into consideration.

The wastefulness of using cottonseed as fertilizer is further evidenced by the fact that when cottonseed is fed to cattle, practically over 90 per cent. of the total fertilizing constituents contained in the seed as meal or hulls are excreted by the animals and thus returned to the soil after having filled their function as feed.

38. Cottonseed Meal.—Cottonseed meal is rich in fertilizing constituents and is extensively used as a fertilizer, its mechanical condition being most excellent for application to the soil. Although cottonseed meal contains large percentages of phosphoric acid and potash, a large proportion of which is in a state readily available to plants, it is used chiefly as a source of nitrogen in fertilizers. The largest proportion of nitrogen in some of the fertilizers sold in the Southern States is derived from this material, as it is usually a cheap source of the necessary nitrogen.

The use of cottonseed meal for fertilizing purposes has increased enormously within the past few years, and at the present time millions of pounds are annually used in the manufacture of fertilizers in the United States. Many cottonseed-oil mills now have, as adjuncts, plants for the complete manufacture of fertilizers.

39. Cottonseed meal alone has been applied with excellent results as a fertilizer for sugar cane, cotton, tobacco, and corn. Its composition with reference to its fertilizing constituents is shown by the following analysis, the average of over two hundred samples being taken: Moisture,

7.60 per cent.; ash, 6.95 per cent.; nitrogen, 7.00 per cent.; phosphoric acid, 2.88 per cent.; and potash, 1.77 per cent. While cottonseed meal, as these facts show, has a high value when applied directly as a fertilizer, the more rational way, as in the case of the raw seed, is to feed the meal to animals, when a large proportion of the fertilizing constituents will be recovered, as previously noted. An additional benefit is also secured in the production of meat, milk, etc.

40. Cottonseed Hulls.—Cottonseed hulls, consisting principally of crude fiber and nitrogen-free extract, are hard and dry and are usually covered with a short, woolly lint. They are not frequently used alone as a fertilizer, as the content of nitrogen is low; but they form a good material to place on clayey lands for the purpose of lightening the earth and giving back a large amount of vegetable matter to the soil. An average analysis of the fertilizing constituents of cottonseed hulls is the following: Moisture, 10.17 per cent.; ash, 2.40 per cent.; nitrogen, .69 per cent.; phosphoric acid, .25 per cent.; potash, 1.02 per cent.; soda, .02 per cent.; lime, .18 per cent.; magnesia, .26 per cent.; sulphuric acid, .08 per cent.; ferric oxide, .03 per cent.; and insoluble matter, .05 per cent.

41. The hulls are usually employed as a fertilizing material in the form of ashes, which vary largely in composition on account of impurities introduced, principally by the use of other fuels in burning them. Most of the potash of cottonseed-hull ashes is in the form of carbonate, a small proportion being present in the insoluble form of silicate, caused by fusion with the sand usually present with the hulls during the incineration. The degree of heat used affects this to a marked degree, from 2 to 5 per cent. being usually present. An average analysis from many samples of hull ashes gave for the principal fertilizing constituents phosphoric acid, 10.85 per cent., and potash, 22.89 per cent. The average moisture for this analysis was 8.95 per cent. The amount of potash is generally between 20 and 30 per cent.

The value of cottonseed-hull ashes depends almost exclusively on the amounts of potash and phosphoric acid they contain. Cottonseed hulls have come into great demand as a tobacco fertilizer, as the cheap source of the requisite potash is here present, and in the form of carbonate is readily available to the plant.

In Table II are grouped the results of the analyses mentioned in the preceding paragraphs. By means of this table, the percentages of the various constituents may be easily compared.

TABLE II

CONSTITUENTS OF COTTONSEED-FERTILIZER MATERIALS

Material	Moisture	Ash	Nitrogen	Phosphoric Acid	Potash	Soda	Lime	Magnesia	Sulphuric Acid	Ferric Oxide	Insoluble Matter
Cottonseed . .	8.42	3.78	3.13	1.27	1.17						
Cottonseed meal	7.60	6.95	7.00	2.88	1.77						
Cottonseed hulls	10.17	2.40	.69	.25	1.02	.02	.18	.26	.08	.03	.05
Hull ashes . . .	8.95			10.85	22.89						

METHODS OF EXTRACTING OIL FROM CAKE

42. Although the process of completely removing the oil from the pressed cottonseed cake has not been adopted to any considerable extent in the United States, the process has for some time been practically demonstrated in Europe as an economical one in many cases. The process has been used for the extraction of oil from some brands of exported American cottonseed cake with great success. It is very economically applied to cake that is to be used as a fertilizer, as, after the extraction of the oil, the material is no longer adapted for feed stuff. The material is, however, more suitable for fertilizing purposes. The solvent used commercially is carbon bisulphide.

43. Carbon-Bisulphide Method.—A vertical section of an apparatus designed for the **carbon bisulphide**

method of extraction is shown in Fig. 4. The boiler for supplying the steam required for the distillation of the carbon bisulphide, and for cleaning out the apparatus, working the pumps, shafting, etc., is erected in a separate building, in order to lessen the risk of igniting the inflammable vapor of the solvent.

At *A* is shown a lead-lined brickwork chamber into which discharge the tube *f*, leading from the condensers *c*, and the pipe *b*, which will be described later. This chamber has a capacity of from 8,800 to 11,000 gallons.

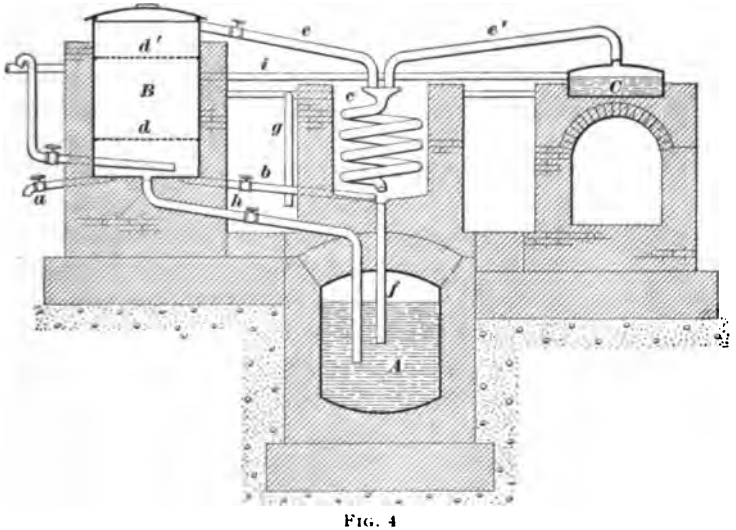


FIG. 4

The extraction cylinder *B* is made of wrought iron and is fitted with a removable cover for charging and emptying the cylinder. It has a capacity of 4,400 gallons and will take a charge of 12 tons of oil cake. About 8 inches above the bottom, it is fitted with a movable perforated false bottom *d*, consisting of wrought-iron plates. The intervening space between the two bottoms is occupied by a perforated steam coil, by means of which steam is blown through the cake after the carbon bisulphide has been drained off.

A second false bottom, composed of a perforated iron disk *d'*, is supported 12 inches from the top of the cylinder

by projections from the walls. Both false bottoms are covered with hemp cloth, and the upper one serves to keep the charge of cake in position. An overflow pipe *i* leads from the side walls of the cylinder into the still *C*, into which it delivers the oil and carbon bisulphide from the first-named vessel.

A gooseneck pipe *e*, located just below the cover of the cylinder, communicates with the condensing coil *c*. Between the false bottom and the actual bottom of the cylinder is a double-branched pipe *h*. This pipe communicates with a pump that, through one of the branches, draws up the solvent from the chamber *A* and forces it by way of the other up through the oil cake in *B*. The extractor can be emptied of residual liquid (solvent) by a tapped pipe *b*, leading into the chamber *A*, after the oil cake has been exhausted of oil, while a tube *a* serves to draw off the condensed water in the bottom of the extractor.

The condensing coil *c*, receiving the contents of the gooseneck pipes *e* and *e'* leading from the extractor *B* and the still *C*, is enclosed in a vessel through which circulates a current of cold water introduced at the bottom and escaping through an overflow pipe *g* at the top. The condensed carbon bisulphide falls from the coil *c* through the pipe *f* into the chamber *A*, from which it is pumped up again for use as a solvent. Evaporation of the bisulphide and the consequent danger due thereto must be guarded against by keeping a supernatant stratum of water in the chamber *A*.

Continuously throughout the operation, the still *C*, capable of holding 1,100 gallons, receives the solution of oil delivered from the extractor by the overflow pipe *i*, and volatilizes the solvent, the oil remaining in the still. The distillation is effected at the outset by a steam coil, steam being afterwards admitted directly to drive off all traces of the carbon bisulphide.

44. When a sample of the liquid issuing from the extractor shows that all the oil has been extracted from the cake, the solvent remaining in *B* is allowed to run back

into the reservoir *A* through the pipe *b*. After all of it has drained away, the portion retained by the cake is driven off by blowing steam in from the jets in the coil under the lower false bottom *d*, so that the vapor of carbon bisulphide is conveyed to the condenser *c* and there liquefied once more, to be collected in the reservoir *A*.

The duration of the extraction is about 12 hours, 2 hours then being allowed for draining. The operation of steaming the cake takes about 8 hours.

The chief cause that has operated to prevent the use of the solvent process for the extraction of oil from vegetable oil cakes has been the difficulty experienced in securing the oil without a characteristic odor; especially so when carbon bisulphide has been used for the extraction. The commercial value of such oils has hitherto been very slight, owing to their acid properties and dark-green color.

45. Tetrachloride-of-Carbon Method.—Consul-General Skinner, of Marseilles, France, transmitted the following to the United States Department of Commerce and Labor in September, 1905, and further commented on this process in the summer of 1906. He says:

"Two well-known concerns in Marseilles have abandoned the naphtha and the carbon-bisulphide processes in favor of tetrachloride of carbon, with results satisfactory to themselves. I am indebted to one of these firms for the following statement, based on actual daily experience:

"The advantages recognized by the use of tetrachloride of carbon as compared with sulphuret of carbon are: (1) The oils obtained are clearer, are free from hyposulphites, and lack the disagreeable odor of oils treated with sulphuret of carbon; and (2) tetrachloride of carbon is not only non-inflammable, but is an extinguisher, eliminating all danger of explosions and fires, so frequent with sulphuret of carbon.

"Tetrachloride of carbon is more expensive than the sulphuret, and its use is relatively expensive, the material employed being more quickly attacked by tetrachloride than by sulphuret.

"Replying to my request for technical information in regard to this process, M. Emile Deiss, the well-known chemist of 3 Rue Suffren, Marseilles, and who is largely responsible for the adoption of the new process commercially, says: Tetrachloride of carbon (CCl_4), chloride of methylene perchlorated, formerly dichloride of carbon, is produced by the action of chlorine on chloroform in the sun. It was discovered by Regnault in 1839.

$$CH + Cl_2 + Cl_2 = HCl + CCl_4$$

"It may be produced also by the reaction of chlorine on ethylene.

"Muller & Dubois, at Reinan, near Manheim, Germany, have obtained a patent for the production of tetrachloride of carbon by the action of chloride of sulphur on sulphuret of carbon in the presence of metals or of metallic chlorides.

"Lever Brothers, of Birkenhead, England, obtained a patent on November 25, 1889, for the extraction of oils by substituting tetrachloride of carbon for sulphuret of carbon, from fatty materials generally—nuts, seeds, oil cakes, etc.—using it in the same manner as sulphuret of carbon and in the same apparatus.

"Advantages: Agreeable odor, non-toxical diluted in the air, absolutely non-inflammable, extinguishes combustion, boils at a temperature sufficiently low not to alter the material being treated, but high enough to avoid appreciable losses, does not decompose nor unite with oils, which, in consequence, have no bad odor; dissolving power considerable; gives an oil that is clearer than sulphuret of carbon. Therefore, it is a perfect dissolvent, offering no danger of intoxication, explosion, or combustion in its manipulation.

"Tetrachloride of carbon is a colorless liquid, which does not unite with water, possesses an agreeable etherized odor; soluble in alcohol and ether; boils at 87° C. Density, 1.629 to 0° C.

"After having enumerated the advantages of tetrachloride of carbon as a dissolvent of fatty matter, it will be easily understood that in a short time it will occupy the place of sulphuret of carbon, which is dangerous to handle, and gives

oils containing sulphur necessarily inferior to oils which are exempt.

"Today tetrachloride of carbon costs about 70 francs ($13.51) per 100 kilograms (220 pounds), and will become cheaper as the industry perfects itself. Taking into consideration the trifling loss in the actual use of this dissolvent, the cost of treating oil cake may now be estimated at 1.50 francs per 100 kilograms (29 cents per 220 pounds), which is very low. Furthermore, oil cake from which the grease has been eliminated by the sulphuret-of-carbon process may be used only as a fertilizer, while oil cake from which the grease has been extracted by means of tetrachloride of carbon may be fed to live stock, therefore commanding a higher price."

The loss of tetrachloride in working is said to be about the same as, or even less than, the amount of the loss of solvent in the sulphuret-of-carbon process. Both processes employ practically the same apparatus.

46. Purification of Extracted Oils.—By a recently devised process, oils extracted by means of carbon bisulphide are completely freed from all objectionable odor and color, and their value and utility thus greatly increased. The process, which is carried out with satisfaction at a good profit, is as follows:

In a cone-bottomed, cylindrical, upright tank capable of holding 10 or 11 tons of oil is placed about 8 tons of the oil to be treated. The tank is about 13 feet high by 6 feet in diameter and is lined throughout with lead $\frac{1}{8}$ inch thick, the joints being soldered together. A cock is placed in the apex of the cone and another in the cylinder, just above the joint of the cone, for drawing-off purposes. Two lead pipes, 2 inches in diameter, descend to the apex of the cone, one for admitting steam, the other for agitating with air, the latter pipe ending in a funnel-shaped aperture.

Eight tons of oil is charged into the receptacle, and to this is added 600 pounds of chloride of lime, stirred to uniform consistency with twice its weight of water. The

whole mixture is vigorously agitated with the blower for several hours; the current of air is then replaced by live steam and 200 pounds of sulphuric acid, diluted with twice its weight of water, is added. This decomposes the lime soap formed by the first operation. When the temperature rises to 100° C., which requires about 5 hours, the steam is shut off and the mixture is allowed to rest. The sulphate of lime and water descend to the bottom of the tank, the separated oil floating above them. The sulphate of lime and water are withdrawn through the lower cock. Through this treatment the oil acquires the color of choice yellow cottonseed oil, but it contains considerable fatty acids liberated by the decomposition of the lime soap. The latter material forms an excellent material for soap making.

47. For other purposes, such as illumination, the acidity must be neutralized entirely. This is accomplished in a tank provided with an ordinary stirrer; the one described in *Cottonseed Oil and Products*, Part 2, may be used for this purpose. The oil is decanted into this tank, and the stirrer is set in motion. A weight of 10° Baumé carbonate of soda, equal to 10 per cent. of the weight of the oil, is now added. After agitating the mixture for about 4 hours, the neutralization is complete. The soap formed by this operation is precipitated by a 10° Baumé solution of common salt. The mixture is again stirred for a couple of hours, when it is allowed to stand at rest for 24 hours. The mixture separates into three layers, the neutral oil at the top, the soap in the middle, and the water at the bottom. The oil, a neutralized oil of good color, is separated and prepared for sale. The soap is collected, and when a sufficient quantity has accumulated, the oil is recovered from it by the treatment described for lime soap.

This process is capable of wide application in the treatment of very low-grade cottonseed or other vegetable seed oils. The employment of upright tanks for both these processes is more advantageous in settling than the use of the shallow tanks ordinarily used in refining.

48. Seed-Oil Separator.—One of the great drawbacks encountered in the process of manufacturing olive and seed oils is the formation of dregs and the subsequent extraction of the oil therefrom, resulting always in a certain amount of waste, no matter what the method of recovering the oil may be. A separator invented by Professor Bracci, of Spoleto, province of Rome, appears to fill a long-felt want. In this apparatus of simple and inexpensive construction, the oil is separated automatically from the water as fast as the mixture from the press runs into the separator.

The separator, in the form of a double cylinder, is filled with fresh water through a tube. The mixture from the press runs into the vat through a sieve and a tube. It becomes diluted and washed in the fresh water, and the oil, being lighter, rises to the surface and runs out through the opening of a cone into the reservoir and out of a valve; meanwhile the surplus water is forced out through a siphon and outlet.

Once in a while it is necessary to renew the fresh water in the separator and to clean the sieve, but all this work can be attended to by a boy without interrupting the work of separation. The separator may be made of any size desired and one single apparatus may be used simultaneously for two or more presses. The great advantage in the use of the separator lies in the facts that a better and more even quality of oil is produced, and that there is no waste.

REGULATIONS OF NEW YORK PRODUCE EXCHANGE

RULES GOVERNING TRANSACTIONS IN COTTONSEED PRODUCTS

49. For the regulation of transactions in cottonseed products among members of the New York Produce Exchange, rules from which the following excerpts are taken, were adopted April 21, 1904, and amended May 18, 1905:

RULE 1. *Sec. II. Oil Arbitration.*—Whenever a case is submitted to the Committee on Oils involving a tender of more than 100 barrels, of different shipping marks, the Committee shall test and give its award on each shipping mark separately, unless both parties to the matter in dispute consent to have the different shipping marks treated as one lot. The Committee shall charge only one fee for each case submitted, irrespective of the number of shipping marks involved, provided, however, that if the Committee is required to pass on each mark separately, no lot shall consist of more than five shipping marks.

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RULE 3. *Sec. I. Fees for Inspectors and Testers.*—When oil or soap stock is sampled by order of the Committee, as prescribed in RULE 19, *Sec. I*, a fee of $2 shall be paid to the Inspector by the party adjudged in fault for the first 100 barrels sampled and 50 cents for each and every 100 barrels or fraction thereof in addition thereto.

Sec. II. Fees for Testing Oil and Soap Stock in Tank Cars.—When oil or soap stock is sampled by order of the Committee, a fee of $2 for each tank car shall be paid to the Inspector by the party adjudged in fault.

Sec. III. Fees for Winter-Oil Tests.—A fee of $2 for each Winter test shall be paid to the Inspector or Tester by the party or parties designated by the Committee.

Sec. IV. Fees for Crude-Oil Tests.—A fee of $3 for each test of crude oil shall be paid by the party or parties designated by the Committee.

RULE 4. *Sec. I. Barrels.*—Oil packages must be good, hardwood, iron-bound barrels, new, or thoroughly cleaned refined-oil barrels, painted or varnished. On delivery of merchantable packages other than the above, an allowance not exceeding 50 cents per barrel shall be made by the seller. Barrels must be delivered in good shipping order, and shall not be under 48 or over 58 gallons each, in case of delivery.

Sec. II. Tares of Barrels.—Tares shall be tested, if required, by either buyer or seller, by emptying 4 barrels of each 100 barrels, to be taken indiscriminately from the lot. Allowance shall be made for difference in tares in excess of 1 pound per barrel. The weighers are required to put a distinguishing mark on the barrels stripped; such mark to be shown on the weight certificate.

RULE 5. *Delivery in Barrels.*—Deliveries of cottonseed oil shall be made by weight at the rate of $7\frac{1}{2}$ pounds net to the gallon.

RULE 6. *Sales f. o. b.*—Unless otherwise specified, all sales of cottonseed products are understood to be f. o. b. Seller cannot be required to deliver less than 100 barrels to any one place, to any vessel, or at any wharf or pier designated by the buyer. The vessel,

wharf, or pier so designated shall be accessible and within the lighterage limits of the Port of New York. It is understood that the seller has fulfilled his contract after he puts the goods within reach of the ship's tackle or lands them on the pier, if required and allowed to do so. The goods shall be delivered free alongside before the expiration of the contract time for delivery.

RULE 7. *Payment.*—All cottonseed products must be paid for in cash, on delivery of the goods. The production of a clean receipt from steamship, warehouse, etc. shall be sufficient evidence of delivery.

RULE 8. *Sec. I. Tank Cars.*—A tank car of cottonseed oil shall be considered as not less than 125 barrels nor more than 160 barrels. Sellers must fill cars to capacity. Any difference in weight, either in excess or shortage, on tank cars shall be settled at the market price on the day of shipment. The date of B / L to determine time of shipment.

Sec. II.—A tank car of cottonseed oil for settlement purposes only, shall be 140 barrels, where no delivery is made. A barrel of oil, if sold loose, is 50 gallons. A gallon of oil is 7½ pounds avoirdupois.

RULE 9. *Sec. I. Tenders.*—All tenders of cottonseed products other than crude and winter oils shall be made between the hours of 10 A. M. and 3 P. M., and, unless rejected before 4 P. M. of the day following, shall constitute a good delivery.

Sec. II.—All tenders of crude and winter cottonseed oils shall be made between the hours of 10 A. M. and 3 P. M., and unless rejected before 4 P. M. of the day following, shall constitute a good delivery.

Sec. III.—All tenders of cottonseed products of a quality superior to that sold shall be deemed a good delivery.

Sec. IV.—If, at the expiration of time allowed buyer for sampling and otherwise testing the goods, as provided for in these rules, the seller demands and the buyer fails to furnish necessary shipping instructions, the seller may proceed to weigh the goods and the buyer must furnish shipping instructions within 24 hours or take delivery of the goods at the point where they were lying when the tender was made, with free lighterage, all risks to be on the buyer.

RULE 10. *Sec. I. Grades of Crude Oil.*—Crude cottonseed oil, to pass as prime, must be made from sound decorticated seed, must be sweet in flavor and odor, free from water and settlings, and must produce prime summer-yellow grade with the use of caustic soda by the best refining methods, with a loss in weight not exceeding 9 per cent. Provided, any oil that refines with a greater loss than 9 per cent., but still makes prime summer-yellow grade shall not be rejected, but shall be reduced in price by a corresponding per cent. of the contract price of the oil. Otherwise, it can be rejected outright.

Sec. II.—Choice crude cottonseed oil must be made from sound decorticated seed, must be sweet in flavor and odor, free from water and

settlings, and shall produce, when properly refined, choice summer-yellow oil at a loss in weight not exceeding 6 per cent. for Texas oil and 7 per cent. for oil from other parts of the country.

Sec. III.—Oil neither choice nor prime shall be called *off oil.* When oil is sold on sample, any oil tendered shall be equal to sample, but if it should refine at a loss exceeding the loss of the sample by not exceeding over 2 per cent., but otherwise equal, it is still a good tender at a reduced price in proportion to the excess loss. The buyer shall have the right to reject the oil outright if it tests beyond 2 per cent. refining loss as compared with the sale sample.

RULE 11. *Sec. I. Grades of Refined Oil.*—Summer-yellow cottonseed oil, to pass as prime, must be brilliant, free from water and settlings, sweet in flavor and odor, and of straw color (not reddish).

Sec. II.—Winter-yellow cottonseed oil, to pass as prime, must be brilliant, free from water and settlings, sweet in flavor and odor, of straw color (not reddish), and must stand limpid at a temperature of 32° F. for 5 hours.

Sec. III. Winter-white cottonseed oil, to pass as prime, must be straw white to white in color, brilliant, sweet in flavor and odor, and must stand limpid at a temperature of 32° F. for 5 hours.

Sec. IV.—Tests for winter cottonseed oil shall be made as follows: A regular 4-ounce sample bottle shall be filled full of the oil to be tested, a thermometer shall be inserted through the cork of the bottle and hermetically sealed. The oil shall then be heated slowly to a temperature not exceeding 80° F., and remain at that temperature not exceeding 15 minutes. It shall then be chilled until it stands at 32° F., at which point it must stand for 5 hours, and must be clear, brilliant, and limpid at the expiration of that time.

Sec. V.—Summer-white cottonseed oil, to pass as prime, must be straw white to white in color, brilliant, and sweet in flavor and odor.

Sec. VI.—Refined yellow oils, designated as "off," may be of inferior flavor, and of a color not exceeding orange, or "reddish," but not "red."

Sec. VII.—Refined yellow oils, designated as "good off," may be off in flavor but must be prime in color.

RULE 12. *Soap Stock.*—Soap stock to be a product of crude cottonseed oil, and all sales thereof, unless otherwise agreed upon by buyer and seller, are made on a basis of 50 per cent. fatty acid, not to fall below 40 per cent. If containing less than 40 per cent. fatty acid, soap stock shall not be considered merchantable. Delivery to be made in iron-bound packages or tank cars.

A contract tank car of soap stock shall be 50,000 pounds, unless otherwise specified.

RULE 13. *Sec. I. Cottonseed Cake.*—A ton of cottonseed cake is 2,240 pounds, unless otherwise agreed. A ton of cracked cake shall be

2,000 pounds, unless otherwise agreed. Cottonseed cake shall be graded and classed as follows:

Sec. II.—Choice cake must be bright yellow in color, sweet in odor, soft and friable in texture, not burnt in cooking, free from excess of hulls, and must produce, when properly ground, choice meal.

Sec. III.—Prime cake must be of good color, yellowish, not brown or reddish, sweet in odor, firm but not flinty in texture, free from excess of hulls, and must produce, when properly ground, a prime meal.

Sec. IV. Off Cake.—All grades of cottonseed cake which are distinctly off in color, taste, or odor, or which have been improperly manufactured, so as to incorporate a very large percentage of lint and hulls, or to produce an exceedingly hard, flinty texture shall be graded as Off Cake.

Sec. V.—Cottonseed cake, unless otherwise specified, shall be packed in good, strong, sound, Dundee bags, either new or second hand, at the option of the seller, unless specified in contract. Packages must be well sewed and in good shipping order, and bear a shipping mark or brand.

RULE 14. *Sec. I. Cottonseed Meal.*—A ton of cottonseed meal is 2,000 pounds, unless otherwise stated. A sack of cottonseed meal is 100 pounds, gross weight. Cottonseed meal shall be graded and classed as follows:

Sec. II.—Choice meal must be the product from choice cottonseed cake when finely ground, and must be perfectly sound, sweet, and of a light yellow (canary) color, free from excess of lint and hulls. Analysis must show at least 8 per cent. ammonia.

Sec. III.—Prime meal must be made from prime cake, finely ground, of sweet odor, reasonably bright in color, yellowish, not brown or reddish, and free from excess of lint or hulls, and by analysis must contain at least 8 per cent. ammonia for meal from Texas and the Mississippi Valley, and 7½ per cent. for meal from the South Atlantic States.

Sec. IV. Off Meal.—Any cottonseed meal which is distinctly deficient in any of the requirements of prime quality, either in color, odor, texture, or analysis or all, is "off."

When off meal is sold by sample, delivery shall equal sample in every respect except in ammonia test, and shall not be rejected if the meal delivered tests not more than one-half of 1 per cent. less ammonia than the ammonia test of the sample sold by, but shall be reduced by a corresponding per cent. of the contract price; otherwise, it can be rejected outright.

Sec. V. Bags.—Cottonseed meal shall be packed in good, sound, Cental or Laplata bags, either new or second hand (except when otherwise stipulated for packages designed for export in kilo or other

bags), 100 pounds, gross weight; which must be well sewed and in good shipping order, and bear a shipping mark or brand.

RULE 15. *Sec. I. Cottonseed Hulls.*—A ton of cottonseed hulls shall be 2,000 pounds.

Sec. II.—A carload of hulls for contract purposes shall be the minimum weight fixed by the railroad tariff prevailing at the point of shipment.

Sec. III.—All claims against shipments shall be as pertaining to all other cottonseed products.

RULE 16. *Sec. I. Cottonseed.*—Cottonseed shall be divided into two classes: *prime* seed and *off* seed.

Sec. II.—Prime seed shall be clean, dry, sound seed, free from dirt, trash, and bolls.

Sec. III. Off Seed.—Seed not coming up to the requirements of prime seed shall be considered off seed. Off or damaged seed shall be settled for on its merits and comparative value as against value of standard prime seed.

RULE 17. *Sec. I. Settlement of Contracts.*—Settlement of contracts for cottonseed oil shall be made on the basis of 53 gallons to the barrel.

Sec. II.—A contract for refined cottonseed oil shall consist of 100 barrels of a total of 5,300 gallons, provided that any deficiency or excess under or above the specified quantity shall be settled for at the bid price of the 3 o'clock call on the day of tender, except on Saturdays, when the bid price of the noon call shall be taken as the settlement price.

Sec. III.—All sales for delivery during a specified month are f. o. b. and at seller's option, unless otherwise agreed.

Sec. IV.—When a seller fails to notify buyer before 4 P. M., two days before the expiration of the month, of his intention to deliver, it shall be deemed a failure of delivery, and the buyer is privileged to buy to cover the contract at the market price of the day following, holding the seller for any difference.

RULE 18. *Sec. I. Sampling Oil and Soap Stock in Barrels.*—When oil or soap stock in barrels is sampled by order of the Committee, the Inspector shall draw samples of not less than 10 per cent. of the lot in question; in the case of oil, samples to be drawn in such a manner as to get a uniform sample and to prevent the introduction of any moisture and excess stearin.

Sec. II. Sampling Oil in Tank Cars.—If in tank cars, at least 2 gallons must be taken well down in the body of the oil, and from this a 1-gallon sample shall be drawn and placed in a perfectly clean tin can, which shall be securely fastened without the use of sealing

wax, and carefully labeled so as to guarantee its identity and correctness and for the use of the Committee.

Sec. III. Sampling Soap Stock in Tank Cars.—When in tank cars, samples shall be drawn in the approximate proportion of 1 pound to each 10 barrels, and a thorough mixture made of same. From the mixture, two 1-pound samples shall be taken and hermetically sealed in a can or jar for the use of the Arbitration Committee.

Sec. IV. Sampling Cake.—Sample pieces not less than 3 inches square shall be taken from at least 5 per cent. of the packages of cake in each carload, or in the entire lot, if not shipped in carload lots, which pieces shall be wrapped in such a manner as to keep each lot separate and distinct and fairly representing the shipment from which taken. The samples shall be sealed and labeled so as to thoroughly identify them and the shipment which they represent. In taking samples the soft edge of the cake, if any, shall first be removed.

Sec. V. Sampling Meal.—Two ounces or more from a sack shall constitute a sample of meal and must be drawn so as to fairly represent the contents of the bag. Twenty samples from each carload, or 50 sacks from each 100 tons, if not shipped in car lots, shall be sufficient to represent a shipment. Separate samples of meal should be well wrapped in heavy oil or waxed paper, sealed or labeled so as to identify them and the shipment they represent. Samples of meal, if of approximately the same grade and quality, need not be kept separate, but may be commingled, in which case they must be placed in a metal mailing or sample box, and carefully marked, showing the number of samples taken, as well as car number and mark.

Sec. VI. Exceptions.—Provided that where large lots of cake or meal are involved, representative samples taken practically as herein prescribed, not less than 5 pounds in weight for cottonseed cake, or 2 pounds for cottonseed meal, shall be deemed a compliance with these rules.

RULE 19. *Sales on Samples.*—When sales are made on sample through a broker, the broker's sample shall be standard. Either party may require such sample to be put in the custody of the Superintendent of the Exchange at the time the sale is made.

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RULE 21. *Fictitious Sales.*—Fictitious sales or false reports of sales are positively forbidden, and will render the parties concerned liable to suspension or expulsion from the Produce Exchange.

RULE 22. *Sec. III. Time of Shipment.*—All trades in cottonseed products shall be either immediate, prompt, or specified dates of shipment: *Immediate* shall be within five working days; *prompt* shall be within ten working days; *specified dates* according to contract. In all cases the bill of lading shall be evidence of date of shipment.

Sec. IV. Buyer's Tanks.—In case the buyer furnishes the tank cars, shipment of same by buyer shall be as follows: Quick shipment of empty tank cars shall be within two working days; immediate shipment of empty tank cars shall be within five working days; prompt shipment of empty tank cars shall be within ten working days.

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Sec. VIII. Seller's Samples.—In case of oil submitted to arbitration on account of quality, the seller's sample of oil, if drawn from the tank car after the tank is loaded, and according to the rules, shall be entitled to consideration of the Committee.

RULE 23. *Special Contracts.*—All transactions in cottonseed oil among members of the New York Produce Exchange shall be governed by the above rules, but nothing therein contained shall be construed as interfering in any way with the rights of members to make such special contracts or conditions as they may desire.

RULE 24. *Changes in Rules.*—No change shall be made in these rules by the Committee on Oils before submitting the same to a meeting of the oil trade, properly called, at which eight members shall constitute a quorum.

REGULATIONS OF INTERSTATE COTTONSEED CRUSHERS' ASSOCIATION

RULES GOVERNING TRANSACTIONS IN COTTONSEED AND COTTONSEED PRODUCTS

50. The following rules, as amended and adopted by the Interstate Cottonseed Crushers' Association, at Atlanta, Georgia, May 15, 16, and 17, 1906, govern transactions in cottonseed products, and are the basis on which the products of the oil mills, such as oil, cake, meal, etc., are sold. They follow closely most of the rules of the New York Produce Exchange as applicable to products, so that uniformity may obtain as far as possible in transactions. They are more specific regarding some things, however, and are therefore given here in full. These rules are enforced among the members of the association and are regarded in general as the usual basis of transactions with outside parties when dealing in these products, as defining in a fair manner all

points to be considered or that would arise in commercial transactions.

The rules of the Texas Cottonseed Crushers' Association conform so closely to those of the Interstate Association that, except for a few unimportant details, they may be regarded as alike.

RULE 1.—In the absence of any special contract to the contrary, the following rules are to govern in all trades in cottonseed and cottonseed products by and between members of the Interstate Cottonseed Crushers' Association.

RULE 2. *Sec. I. Cottonseed.—Prime cottonseed* shall be clean, dry, and sound, free from dirt, trash, and bolls.

Sec. II. Off Seed.—Cottonseed not coming up to the requirements of prime seed shall be considered off seed. Off or damaged seed shall be settled for on its merits and comparative value as against value of standard prime seed.

RULE 3. *Cottonseed Oil.*—A tank car of cottonseed oil for contract purposes shall be 125 barrels, unless otherwise specified. A barrel of oil, if sold loose, is 50 gallons. A gallon of oil is 7½ pounds avoirdupois.

RULE 4. *Sec. I. Crude Oil.*—Crude cottonseed oil may be sold either loose or in barrels, as agreed between seller and buyer. If in barrels, they shall be good, new, iron-bound barrels, properly silicated, or thoroughly steamed and cleaned refined-petroleum barrels. Packages must be in good shipping order, and contain not less than 48 gallons each, provided that the aggregate of delivery on any sale shall equal 50 gallons for each barrel sold. On delivery of other than above barrels, an allowance of 50 cents per barrel shall be made by seller.

Sec. II.—Choice crude cottonseed oil must be made from sound decorticated seed; must be sweet in flavor and odor; free from water and settlings; and shall produce, when properly refined, choice summer-yellow oil at a loss in weight not exceeding 6 per cent. for Texas oil and 7 per cent. for oil from other parts of the country.

Sec. III.—Prime crude cottonseed oil must be made from sound decorticated seed; must be sweet in flavor and odor; free from water and settlings; and must produce prime summer-yellow oil with the use of caustic soda by the best refining methods, with a loss in weight not exceeding 9 per cent. Provided, that any oil that refines with a greater loss than 9 per cent., but still makes prime summer-yellow oil, shall not be rejected, but shall be reduced in price by a corresponding per cent. of the contract price of the oil. Otherwise, it can be rejected outright.

Sec. IV. Off Crude Cottonseed Oil.—Oil neither choice nor prime shall be called "off" oil. When off oil is sold by sample, any oil tendered shall equal sample, but if it should refine at a loss exceeding the loss of the sample by not over 5 per cent. but otherwise equal, it is still a good tender at a reduced price in proportion to the excess loss.

The buyer shall have the right to reject the oil outright if it tests beyond 5 per cent. refining loss as compared with the sale sample.

RULE 5. *Tank Bottoms.*—Sales and deliveries of tank bottoms, to be a product of crude cottonseed oil, shall be a matter of contract between seller and buyer, and may be bought and sold by sample, or otherwise, as may be agreed upon.

RULE 6. *Sec. I. Refined Oil.—Choice summer-yellow cottonseed oil* must be sweet in flavor and odor, clear and brilliant in appearance, and free from moisture.

Sec. II.—Prime summer-yellow cottonseed oil must be clear, sweet in flavor and odor, free from water and settlings, and of no deeper color than 35 yellow and 7.1 red on Lovibond's equivalent color scale.

The color examination shall be made as follows: The oil is placed in a pure white 4-ounce sample bottle; the depth of the oil in the bottle shall be $5\frac{1}{4}$ inches. The bottle shall be placed in a tintometer which is protected from any light, except reflected white light, and the reading made at the temperature of about 70° F. If the oil is of deeper color than the glass standard, 35 yellow, 7.1 red, it shall not be prime.

Sec. III.—"Good off" summer-yellow cottonseed oil may be off in flavor, and / or odor, but must be prime in color.

Sec. IV.—Off summer-yellow cottonseed oil shall be free from water and settlings, off in taste and color, and should be sold by sample.

Sec. V.—Settlements of contracts for refined cottonseed oil shall be made on a basis of 53 gallons to the barrel. Packages for refined oil must be good, hardwood, iron-bound barrels, new or thoroughly cleaned refined cotton-oil barrels, painted or varnished. They must be delivered in good shipping order, and shall not be under 50 or over 60 gallons each in case of delivery. On delivery of packages other than as above, an allowance not exceeding 50 cents per barrel shall be made by seller. Tares shall be tested, if required by either seller or buyer, by emptying 4 barrels of each 100 barrels, to be taken indiscriminately from the lot. Allowance shall be made for difference in tares in excess of 1 pound per barrel.

RULE 7. *Soap Stock.*—Soap stocks to be a product of crude cottonseed oil, and all sales thereof, unless otherwise agreed upon by seller and buyer, are made on a basis of 50 per cent. fatty acid, not to fall below 40 per cent. If containing less than 40 per cent. fatty acid, soap stock shall not be considered merchantable. Delivery to be made in iron-bound, hardwood packages or tank cars.

A contract tank car of soap stock shall be 50,000 pounds, unless otherwise specified.

Nothing in this rule shall be interpreted to fix the amount of draft to be drawn against shipments of soap stock.

RULE 8. *Sec. I. Cottonseed Cake.*—A ton of cottonseed cake is 2,240 pounds, unless otherwise specified.

A ton of cracked cottonseed cake shall be 2,000 pounds, unless otherwise specified.

Cottonseed cake shall be graded and classed as follows:

Sec. II.—Choice cottonseed cake must be bright yellow in color, sweet in odor, soft and friable in texture, not burnt in cooking, and free from excess of hulls.

Sec. III.—Prime cottonseed cake must be of good color, yellowish, not brown or reddish, sweet in odor, firm but not flinty in texture, and free from excess of hulls.

Sec. IV. Off Cottonseed Cake.—All grades of cottonseed cake which are distinctly off in color, taste, or odor, or which have been improperly manufactured, so as to incorporate therein a very large percentage of lint and hulls, or to produce an exceedingly hard, flinty texture.

Sec. V. Cake Packages.—Cottonseed cake, unless otherwise specified, shall be packed in good, strong, sound bags, either new or second hand, at the option of the seller, unless specified in contract. Packages must be well sewed and in good shipping order, and bear a shipping mark or brand.

In the case of shipment of carload lots or over, the Official Port Inspector or some Public Weigher, after delivery to buyer may reweigh the shipment (but if weighed on track scales, actual gross, tare, and net weights must be given), and certificates so taken and properly sworn to shall determine weight in all cases where cake is sold "delivered" or "weights guaranteed at destination," provided the shipment is not broken in transit.

In case of loss in weight, the expense of weighing shall be paid by the seller, but in case the weights are found to be correct or underweighed, the cost shall be paid by the buyer, and the seller shall be paid for the excess weight so determined.

RULE 9. *Sec. I. Cottonseed Meal.*—A ton of cottonseed meal is 2,000 pounds, unless otherwise stated. A sack of cottonseed meal is 100 pounds, gross weight.

Cottonseed meal shall be graded and classed as follows:

Sec. II.—Choice cottonseed meal must be finely ground, perfectly sound, and sweet in odor, yellow, free from excess of lint and hulls, and, by analysis, must contain at least 8 per cent. of ammonia.

Sec. III.—Extra prime cottonseed meal must be finely ground, of sweet odor, reasonably bright in color, yellow, not brown or reddish, and, by analysis, must contain at least 7½ per cent. of ammonia.

Sec. IV.—Prime cottonseed meal must be finely ground, of sweet odor, reasonably bright in color, and, by analysis, must contain at least 7 per cent. of ammonia.

Sec. V. Off Cottonseed Meal.—Any cottonseed meal which is distinctly deficient in any of the requirements of prime quality, either in color, odor, texture, or analysis, or all, shall be deemed off meal and should be sold by sample.

Sec. VI.—Cottonseed meal not coming up to contract grade shall be a good delivery if within one-half of 1 per cent. of the ammonia contents of the grade sold, or the sale sample, but the settlement price shall be reduced at the rate of one-tenth of the contract price for each 1 per cent. and proportionately for the fractions, of deficiency in ammonia.

Sec. VII. Weights and Packages for Cottonseed Meal.—Cottonseed meal shall be packed in good, sound Cental or Laplata bags, either new or second hand, 100 pounds, gross weight (except where otherwise stipulated for packages designed for export in kilo or other bags). Bags must be well sewed and in good shipping order, and bear a shipping mark or brand.

Sec. VIII. Reweighing.—On shipments of carload lots or over, the official Port Inspector or some Public Weigher shall have the right to open cars after delivery to buyer and take at random therefrom and reweigh a number of bags equal to 5 per cent. of the entire number in the car, and, in case a shortage is found, then the entire contents of the car shall be reweighed. In case a loss is shown, the expense of weighing shall be paid by seller; but, in case the weights are found to be correct or underweighed, the cost shall be paid by buyer.

RULE 10. *Rejections of Cottonseed Cake and Meal.*—Should the whole or any portion of a shipment of cottonseed cake or meal not turn out equal to the contract quality, the buyer shall take delivery at an allowance to be fixed by arbitration, but if any portion shall be adjudged not to be within $1.50 per long ton of the value of contract quality, the buyer shall have the option of rejecting and invoicing back such portion at market price of the quality contracted for on the day of the rejection, the market price to be decided by the arbitrators.

RULE 11. *Sec. I. Linters.*—Cottonseed linters shall be governed in sale by special contract.

Sec. II.—Mill run linters shall be made from reginning cottonseed, without regard to grade, and shall be free from flues, or lint obtained from thrashed seed or grabots.

Sec. III. Sales of Linters.—When a sale is made of season's or balance of season's output of linters, the seller must ship and the buyer must receive all the linters seller makes to the end of the season, provided, that when estimated number of bales is stated in contract, or in confirmation of sale or purchase, the buyer may demand and

seller must ship, or may ship whether demanded or not, 15 per cent. in excess of estimated quantity if he makes a sufficient number of bales to enable him to do so, and buyer must receive and pay for same at contract price. Should seller not make the quantity estimated, he shall deliver the number of bales made, and shipment of 85 per cent. of the estimated quantity shall be deemed a fulfilment of the contract.

Sec. IV. Weights and Packages for Linters.—A bale of linters for contract purposes is 500 pounds, gross weight, with a maximum or minimum allowance of 5 per cent. Bales weighing less than 350 pounds may be rejected by buyer.

Linters must be well covered, with an average of 6 metal ties per bale.

RULE 12. *Sec. I. Hulls.*—A ton of cottonseed hulls shall be 2,000 pounds.

Sec. II.—A carload of hulls for contract purposes shall be the minimum weight fixed by the railroad tariff at point of shipment.

Sec. III.—All claims against shipments shall be as pertaining to all other cottonseed products.

RULE 13. *Sec. I. General Rules.*—All offers, sales, or purchases of cottonseed products shall be understood, unless specified to the contrary, to be f. o. b. cars at the mill, weights and quality guaranteed at destination when received in original packages in good order, loss or damage by accident or wreckage in transit to be at buyer's risk. Unless specially stated otherwise, oil shall be considered as sold loose, and buyer shall furnish tank cars.

Sec. II. Trades by Telegraph.—On all trades by telegraph, day messages requiring day answers shall be open until 12 o'clock midnight of the day on which sent; night messages shall be open until noon following the night on which sent. The time when telegrams are filed in telegraph office sending same to govern, and this section to apply when no specific time is stated in the original offer.

Sec. III. Time of Shipment.—All trades in cottonseed products shall be for either immediate, prompt, or specified dates of shipment.

(1) *Immediate* shall be within 5 working days.

(2) *Prompt* shall be within 10 working days.

(3) *Specified dates* according to contract.

In all cases, the bill of lading shall be evidence of the date of shipment.

(4) It is understood that this rule does not refer to or in any way affect the sale of oil in buyer's tanks.

Sec. IV.—All sales of cottonseed products, unless otherwise specified, shall be for cash, payment to be made by resident buyers on presentation of invoice with railroad ticket signed, or bill of lading attached showing delivery of goods to the carrier in good order.

Unless specially stipulated, payment of non-resident buyers shall be by sight or demand draft at contract price, with not exceeding one-quarter of 1 per cent. exchange, with bill of lading attached, showing delivery of goods to the carrier in good order, unless otherwise agreed.

Sec. V.—Any tender of a grade of oil, meal, cake, or linters better than the grade sold shall be deemed a good delivery.

Sec. VI.—When goods are delivered to the carrier as agreed, whether in whole or partial completion of trade, payment for same shall become due, if presented during banking hours, and all risks belong to the buyer.

Sec. VII.—On all sales of cottonseed products to or through regular brokers, the sellers shall pay the brokerage, unless otherwise specially agreed.

Sec. VIII.—When a trade is closed with or through a broker, it shall be understood that his fee has been earned, whether the goods are finally delivered or not.

Sec. IX. Buyer's Tanks.—In case the buyer furnishes tank cars, shipment of the entire number of tank cars contracted for shall be made by the buyer as follows:

(1) *Immediate shipment* of empty tank cars shall be within 2 working days, date of contract not included.

(2) *Quick shipment* of empty tank cars shall be within 5 working days, date of contract not included.

(3) *Prompt shipment* of empty tank cars shall be within 10 working days, date of contract not included.

(4) *Under specified shipments*, tank cars shall be forwarded by buyer in such time that under the ordinary course of transportation they shall reach the seller in time to allow him to make delivery as per contract.

(5) Buyer shall notify seller when tank cars are shipped, giving location of car, with number and name, and follow up such notice with railroad receipt or bill of lading. Railroad records shall determine date of shipment.

Seller shall, in like manner, give notice of arrival of tank car and reshipment of same, sending railroad receipt or bill of lading, the date of which shall be evidence of shipment of loaded car.

(6) Failure on the part of buyer to ship cars in the proper time and give due notice thereof shall entitle the seller, at his option, to cancel the contract or exact demurrage at the rate of $2 per tank car for every day's delay, or, upon notice, the right to sell the oil for the account of buyer, after the lapse of 10 days, at the best obtainable price through any cotton oil broker in good standing, holding the buyer for losses. Sale to be for earliest obtainable shipment, original buyer to pay demurrage for all delays. But in case it is shown that the tank cars were shipped in due time, as above specified, and delayed en route, the seller must fill them, charging the buyer $2 per day per tank car,

as above specified, and the buyer must accept them under the contract. In case the seller elects to cancel, he must, after he has acquired the right to cancel any contract, or part thereof, notify the buyer, by registered letter and wire, of his intention to do so. Failure to give such notice shall operate as a renewal of the contract and shall extend the time of same by as many days as seller allows to lapse before giving notice.

(7). In case of immediate shipment, in case of quick shipment, and in case of prompt shipment, buyer cannot be held responsible for railroad delays, and no demurrage shall accrue.

In case of cars delayed for specified shipment (paragraph 4) the seller may charge the buyer $2 per car demurrage for each day which elapses after expiration of contract time. In case a tank car is disabled or lost, another tank car shall be forwarded immediately by buyer.

(8) It is understood that the arrival of tank cars at the town where mill or refinery is located shall constitute delivery, as above.

(9) It is understood that nothing in the above rule may be taken to limit or interfere with the measure of damages that may arise under the contract.

Sec. X.—Seller shall in all cases load tank cars within 48 hours of arrival at destination, and to their full capacity, when within contract requirements. In case the seller does not load the tank cars within 48 hours after arrival, he shall pay the buyer $2 for each tank car for every day's delay beyond 48 hours (in this case "destination" means "mill" when within free switching limits of the town where mill is located) or if, after the lapse of 10 days after the expiration of contract time of shipment, the tank cars have not been loaded, the buyer shall have the right to purchase the quantity of oil due on contract for the account of seller at the lowest obtainable price through any cotton oil broker in good standing, holding the seller for loss sustained.

Tank cars delayed during settlement of disputes by arbitration or otherwise shall be subject to demurrage at the rate of $2 per day, less the customary unloading time of 48 hours, the party in error to pay the demurrage. Seller shall, in all cases before loading, inspect tank cars and clean them, if necessary, at the expense of the buyer, charging him actual cost for same.

Sec. XI. Time Contracts.—When cottonseed products other than oil are sold for shipment over one or more months, it shall be at seller's option as to the time in each of the months named as to shipment, but the seller shall ask buyer for shipping orders at least 10 days before he shall make shipment. Sales for shipment over two or more months are understood to be for equal shipments during each month named, unless otherwise specified in the contract.

RULE 14. *Sec. I. Claims.*—The place of fulfilment of contracts for export is to be at American seaport of shipment or border.

Sec. II.—All claims against shipments of cottonseed products must be made within 10 days after their arrival at American points of destination (except claims for demurrage on tank cars, in which case 30 days shall be allowed in which to file claims, and except products for export, in which 30 days shall be allowed after arrival at American border or port).

Sec. III.—No claims from any foreign market will be recognized unless the proper samples of the goods are taken and preserved previous to their leaving American shore. But on shipments made on through bill of lading only, samples drawn as provided in the rules governing samples before removal of goods from foreign dock will be recognized.

Sec. IV.—Where claims are made and not sustained, the claimant must pay all expenses incurred.

Sec. V.—All claims to be brought before the Arbitration Committee of this Association must be accompanied by an affidavit from a reliable party, substantially in the following form, describing and identifying the sample submitted as taken from and fairly representing the entire shipment.

Sec. VI. Form of Claims.—I, the undersigned, do hereby make affidavit that I have drawn fair and true samples from _____ packages of _____ being not less than _____ per cent. of the entire number of packages embraced in a shipment made by _____ from _____ as evidenced by bill of lading dated _____ and issued by _____.

The samples were carefully taken so as to secure a fair representation of the contents of the individual package and a true average of the quality of the entire shipment.

I certify to the correctness of the samples, which are marked as follows: _____ and which represents the shipments marked or identified as follows; _____ or contained in _____.

Sworn to before me, a notary or justice of the peace of _____ County, and State of _____ and duly authorized by law to take depositions, this _____ day of _____ 190__.

RULE 15. *Sec. I. Weights.*—(1) A claim for loss in weight, to be entitled to consideration, must be supported by the sworn certificate of the Association's Official Inspector or a Public Weigher at point of destination.

(2) In the case of oil, however, the oil shall be weighed by a sworn Public Weigher at destination, and his certificate furnished immediately to both seller and buyer. It must show condition of tank

car, and, if weighed on track scales, the gross, tare, and net weights. Also that car was uncoupled and free while being weighed. If on tank scale, the certificate must show the condition of both tank car and tank scale, the thorough emptying of same, and the condition of all connecting pipes.

(3) All expenses of weighing and inspection to be paid by the buyer.

(4) In case of weights of oil submitted to arbitration, the seller's proof of weights at the mill shall be entitled to consideration by the Arbitration Committee.

RULE 16. *Sec. I. Quality.*—In case of oil submitted to arbitration on account of quality, the seller's sample of oil, if drawn from the tank car after the tank is loaded, and according to the rules, shall be entitled to consideration by the Arbitration Committee.

Sec. II.—Where sales are made for shipment in buyer's tanks, and the oil is rejected on delivery because not up to the contract, the seller, on being notified, should, within 5 days thereafter, dispose of the oil through any recognized broker on the open market for the account of whom it may concern. If the seller fails to make disposition of said oil within 10 days, the buyer shall take possession of or sell the oil for account of whom it may concern, and buyer's claims shall be decided by the Arbitration Committee, if seller and buyer cannot agree. In the case of arbitration of oil rejected under this article, the Arbitration Committee shall assess the actual loss and damages against the loser of the arbitration.

RULE 17. *Sec. I. Samples.*—Samples shall in every case be drawn in the presence of representatives of both seller and buyer, at American destination, by a reliable party or parties, who shall make affidavit as prescribed by these rules in the "Form of Claims." (RULE 14, *Sec. VI.*)

Sec. II.—If the seller refuses or neglects for 48 hours after notification to appear in person or appoint a representative to draw the samples in the presence of the buyer or his representives for arbitration, then the buyer may appoint any disinterested person to draw such samples. This is to apply to all cottonseed products.

Sec. III.—Samples drawn and presented to the Association, with all expenses paid in accordance with the above requirements and with the prescribed form and agreement attached, shall be considered sufficient evidence for arbitration.

Sec. IV.—Samples representative of any shipment of cottonseed products, to secure the official recognition of this Association or its committees, must be secured in substantially the following manner:

Sec. V.—In case of contention, and when agreed samples are not furnished, the Arbitration Committee shall consider samples furnished by both the seller and the buyer, careful attention being given to

properly sworn statements as to the manner of procurement and identification of the samples furnished.

Sec. VI.—If oil is in tank cars, at least 2 gallons must be taken well down in the body of the oil, and from this a 1-gallon sample shall be drawn and placed in a perfectly clean tin can, which shall be securely fastened without the use of sealing wax, and carefully labeled, so as to guarantee its identity and correctness, and for the use of the Arbitration Committee.

Sec. VII.—When oil in barrels is sampled by order of the Arbitration Committee, samples shall be drawn from 10 per cent. of the barrels selected at random, each sample to be taken from a separate barrel, so as to represent its entire contents, and drawn in such manner as to prevent any introduction of moisture; each sample so taken shall be sealed and labeled, as above provided for.

RULE 18.—When in tank cars, soap-stock samples shall be drawn from flowing stock at regular intervals as tank is being loaded, in the presence of a representative of the seller, as provided in RULE 17. Samples shall be taken in the approximate proportion of 2 pounds to each 10 barrels and a thorough mixture made of same. From this mixture three 1-pound samples shall be taken, which shall be hermetically sealed in can or Mason jar with rubber gasket; the first to be forwarded to the buyer, the second to be retained by the seller and tested by his chemist, and the third to be retained intact, hermetically sealed, and properly marked for identification by the seller. If impractical for the buyer to be represented when samples are drawn at the mill, samples shall be drawn by the buyer at destination in the presence of a representative of the seller or by a Public Inspector. In the event of difference in the test between the seller's chemist and the buyer's chemist, the third sample shall be submitted to a disinterested chemist to be agreed upon.

If in barrels, samples shall be drawn with a trier from each and every barrel and from this mixture a gallon sample be taken which shall constitute the test for the lot, samples to be drawn from tanks or barrels by sampler.

RULE 19. *Cake.*—Sample pieces not less than 3 inches square shall be taken from at least 5 per cent. of the packages in each carload, or in the entire lot, if not shipped in carload lots, which pieces shall be wrapped in such manner as to keep each lot separate and distinct, and fairly representing the shipment from which taken. These samples shall be sealed and labeled so as to thoroughly identify them and the shipment which they represent.

RULE 20. *Meal.*—Two ounces or more from a sack shall constitute a sample of meal, and must be drawn so as to fairly represent the entire contents of the bag. Twenty samples from each carload, or

50 sacks from each 100 tons, if not shipped in car lots, shall be sufficient to represent a shipment. Separate samples of meal should be well wrapped in heavy paper, sealed and labeled, so as to identify them and the shipment they represent. Samples of meal, of approximately the same grade and quality, need not be kept separate, but may be commingled, in which case they must be placed in a metal mailing or sample box and carefully marked, showing the number of samples taken, as well as car number and mark.

Provided, that where large lots of cake or meal are involved, representative samples taken practically as herein prescribed, not less than 5 pounds in weight for cottonseed cake, or 2 pounds for cottonseed meal, shall be deemed a compliance with these rules.

RULE 21. *Arbitration.*—(1) Arbitrations may be held at New York, Memphis, Atlanta, New Orleans, Chicago, Dallas, and Galveston, as agreed by the parties at difference, and in case they cannot agree, then as determined by the Secretary of the Association. At the above-mentioned points, and all others where application is made by not less than ten members, and if in his opinion it is advisable, the President of this Association, as soon as convenient after his election, shall appoint a Permanent Committee of Arbitration to consist of five members, any three of whom shall constitute a quorum for the transaction of business. Each committee shall meet upon call of its Chairman, as often as is necessary for the prompt despatch of business, and, as compensation, shall receive for each case decided at such meeting a fee of $15, to be divided as later provided, together with traveling and hotel expenses of the members actually in attendance and serving.

(2) The party demanding the arbitration, at the time of the demand, and the other party, on consenting to it, shall deposit with the Secretary of the Association a fee of $25, of which, in case of loss, there shall be paid for each case $10 to the Association, $15 to the Committee of Arbitration, which amount shall be divided equally between the members of the committee actually serving on the case, who shall also receive, in addition, their expenses, as above provided. Chemist, if any, to be paid by loser.

(3) No personal appearance will be permitted before the Arbitration Committee except upon the unanimous request of the Committee. Before calling the Arbitration Committee together, the Secretary of the Association shall procure from each party to the arbitration an agreement in writing to abide by the decision of the Arbitration Committee, and to pay immediately the amount of the award.

(4) An appeal to the Executive Committee from the decision of the Arbitration Committee may be had by either party on written notice within 5 days in cases where the award is over $300. The party making the appeal shall deposit with the Secretary of the Association the full amount of the award, plus $50, to cover expenses of the appeal.

(5) In cases of an appeal, the Secretary shall call the Executive Committee together, and the majority of them shall constitute a quorum, and each member serving shall receive $10 and his traveling expenses to and from the place of meeting. The loser shall pay all expenses of the arbitration and the traveling expenses of the arbitrators.

(6) Should a member fail or refuse to submit to the demand of another member for arbitration, or delay or obstruct such demands for 5 days after proper notice, the Chairman of the Permanent Committee on Arbitration, on receipt of such complaint, shall proceed at once to satisfy himself as to the facts, and, these being satisfactory, shall immediately proceed with the arbitration ex parte, and the decision so rendered shall be of full force and effect.

(7) Should any member of this Association refuse to submit his differences to arbitration under these rules, or, after an ex-parte hearing has been had, refuse to pay in full any award against him of the Arbitration Committee, the other member or members to the arbitration may report the matter to the Chairman of the Permanent Committee on Arbitration, who shall at once proceed to satisfy himself as to the correctness of the complaint, and, if confirmed, shall at once notify the President of the Association, who shall immediately, through the Secretary, demand of the party at fault full compliance with the requirements of these rules within 5 days thereafter, and if the member fails to comply with such official demand he shall be immediately expelled from the Association, and the President, over his signature, countersigned by the Secretary, shall so notify him, and at the same time and in the same manner, shall issue a circular letter to every member of the Association notifying them that such member has been expelled from the Association for not conforming with the provisions of RULE 21.

Any member so expelled shall not again become a member of this Association until such claim shall have been satisfied in full and then only upon a majority vote of the Executive Committee.

(8) All expenses of an arbitration shall be borne by the party against whom award is made, and whether such award is for the full amount claimed by the other party or for only a part of same. In ex-parte cases, should the losing member fail to pay the award and expenses, the party demanding such procedure shall stand the arbitration expenses.

(9) Should any seller or buyer incorporate in any contract of purchase and sale conditions looking to the adjustment of differences that may arise under it by any other tribunals than those provided by this Association, and existing under and governed by its rules, it is understood that such contract is made and accepted entirely independent of this Association, and differences which arise under it shall not be subjects for its consideration or arbitration.

(10) Any member with whom an arbitration is demanded shall not be allowed to resign before all matters in question are settled, the Secretary to give all notices required under this rule by registered mail.

RULE 22. *Transactions in Cottonseed Products.*—All transactions in cottonseed products among the members of this Association shall be governed by the above rules; but nothing herein contained shall be construed as interfering in any way with the rights of members to enter into specific written contracts stating special conditions. Either party to a trade may demand a formal written contract as soon as the trade is completed. Such contract, unless especially excepted, being subject to all the rules of this Association.

RULE 23. The foregoing rules shall apply on all contracts made on and after August 1, 1906, on which date they shall become effective, superseding all rules heretofore in effect.

ANALYTICAL METHODS APPLICABLE TO COTTONSEED PRODUCTS

PHYSICAL AND CHEMICAL TESTS

51. Introduction.—In the examination of a cottonseed oil, the first tests invariably applied are of a purely physical and superficial character, consisting of the examination by sight, smell, and taste. Whether or not further supplemented by analytical methods, these tests are very essential. Although apparently simple to those familiar with cottonseed oils, to the person desiring to perfect his knowledge and judgment of cottonseed products, these tests are of the greatest importance. The cultivation of judgment should be such that when these tests are applied they will give a correct idea of the grade and class of oil under examination. It is only by practice that this knowledge can be obtained, and primarily it must be applied by comparison with standard samples of the different grades of oils. While frequently to the practical oil miller the science of applied chemistry does not appeal, the preceding tests do and are always applied in his working of cottonseed oil. When supplemented by the usual chemical tests, the grading of cottonseed oils is comparatively easy.

52. The methods of analyzing cottonseed products are along the general lines already considered. Many of the determinations given in *Quantitative Analysis* can be applied to such products as nitrogen (ammonia), phosphoric acid, potash, etc. in cake and meal. Others, which are especially applicable to certain cottonseed products, will be presented, among them being the methods for ascertaining the amount of crude oil left in the foots, the percentage of oil left in the cottonseed cake after pressing, the amount of free fatty acids in the oil, etc. These tests are of the greatest importance in the cottonseed-oil industry, for it has been through their application that great economic changes have taken place in recent years in oil milling and refining. By the application of analytical chemistry, the methods and processes of manufacturing are reduced to a scientific basis—the key to success in this, as in all other similar industries.

The manufacture of cottonseed oil and other products is constantly being improved, largely through the control of the operations exercised by the application of analytical methods.

53. Cottonseed oil is representative of a class group, occupying a position midway between the drying vegetable oils, as represented by linseed and similar oils, and the non-drying vegetable oils, of which olive oil is a representative. It is classed as a semidrying oil, acquiring, after long exposure in a thin film, the property of drying. The characteristics of cottonseed oil, as observed in regard to the various chemical reactions, coloration tests, etc., also occupy an intermediate position between the two groups mentioned. More detailed information regarding this matter will be found in *Quantitative Analysis*.

The following are some of the observed analytical data taken from various sources relating to cottonseed oil and the fatty acids therefrom:

Crude oil, specific gravity	.916 to .920
Refined oil, specific gravity . . .	.922 to .926
Refined oil, solidifying point . . .	1° C. to 10° C.

Free fatty acids	.4 to . . .
Iodine number	106 to 112
Maumene's test	80 to 90
Saponification value (mmg. *KOH*)	201.6 to 208
Iodine number	111 to 115.7
Unsaponifiable matter	.90 to 1.36
Fatty acids from cottonseed oil:	
Specific gravity at 99° C. (water at 15.5° C. = 1)	.8467
Specific gravity at 100° C. (water at 100° C. = 1)	.8816
Titer test	32.2° to 37.6°
Melting point	35° to 40°

54. Determination of Free Fatty Acids in Oils. The quantity of lye required for the refining of any oil depends on the percentage of free fatty acids present, or as much as it is desired to extract, all in excess of this being absolute loss. If too much caustic-soda solution is used, in addition to saponifying the free fatty acids, it is liable to cause a saponification of the oil itself and thereby cause a large shrinkage. This is most likely to occur at high temperatures. Further, any excess of lye remaining in the oils treated is lost and wasted, as it must subsequently be removed in the after treatment.

Commercially, the free fatty acids of vegetable and animal oils are all calculated as oleic acid, although other acids, such, for example, as rosin acids, may be present in small proportions. For technical purposes, however, such calculation of the free fatty acids is sufficiently accurate. The determination of these acids is carried out by titrating an alcoholic solution of the free fatty acids with a standard caustic alkali, using phenol phthalein as an indicator. The solutions required are: (*a*) a neutralized, dilute alcohol (ethyl or purified methyl), (*b*) an alcoholic solution of phenol phthalein, and (*c*) a standard caustic alkali, either seminormal or decinormal. The former is preferable in ordinary working, and is convenient in a commercial way. The alcohol is

neutralized by adding to it, drop by drop, a weak caustic solution until it retains after violent shaking the light-pink tint occasioned by neutralization of the acid present in the alcohol. The usual indicator, phenol phthalein, is added before the caustic is added. Solutions (*a*) and (*b*) may thus be combined, as is usually done for convenient working. The alcohol may be diluted with an equal volume of distilled water.

55. About 50 grams of oil, or any known weight, is placed in the usual 4-ounce sample bottle, and from 60 to 80 cubic centimeters of the neutralized alcohol is added to it. The contents are well shaken together, placed on the water bath or in hot water, and heated to about 180° to 190° F. The oil and alcohol are well shaken again to effect the complete solution of the fatty acids. With most cottonseed oils the color will always disappear, as the usual grades have naturally more or less free acid. The seminormal solution of caustic soda or potash is now added, small portions at a time, until the pink color caused by the caustic disappears very slowly. At this period, the seminormal solution is added drop by drop until after violent shaking of the sample the pink coloration remains to the alcohol. The end reaction is very sharp and delicate, and the point of neutralization is easily seen in light-colored oils. With crude oils, however, it is necessary to proceed near the finish very cautiously, as the dark color of the oil is apt to mask the end point, causing an excess of caustic to be added. This may be obviated by allowing the contents of the bottle to stand a few moments, when the oil and alcohol will separate, and the color of the latter may then be clearly observed. An example of titration will make this determination clear. It required for 50 grams of oil 5.5 cubic centimeters of seminormal alkali to produce the required pink coloration. As 1 cubic centimeter of this solution corresponds to .141 gram of oleic acid, there must be $5.5 \times .141 = .7755$ gram of oleic acid in the 50 grams of oil taken. Hence, $.7755 \div 50 = .0155$, or 1.55 per cent. of free fatty acids in the sample titrated.

56. Test for Mineral Acids in Oil.—Mineral acids are likely to be present in cottonseed oils only after refining by an acid process. When the presence of mineral acid is suspected, the oil is agitated with warm water and the aqueous liquid separated. The latter is then tested by adding to it a solution of methyl orange, which will give the usual red or orange coloration if any mineral acid is present.

57. Color Test for Refining Crude Oil.—The solution required for the color test is a 20° Baumé caustic-soda solution. Into a large oil-sample bottle 100 cubic centimeters of the crude oil is placed and a few cubic centimeters of the solution added. As the color test gives the key to the probable, if not actual, amount of caustic soda to be used in refining, it is performed by adding trial quantities until the desired color in the sample is obtained. To the quantity of oil in the bottle, 2 cubic centimeters of the solution as a trial is added and the whole well shaken together for 1 minute. The bottle and contents are then placed in hot water at 160° F. for 5 minutes. The oil is then filtered into another bottle and compared with a similar bottle of prime yellow oil as a standard. If the color of the filtered oil is not light enough, another test is made in the same manner, using this time 2.25 cubic centimeters of caustic solution. These tests are continued until the standard prime yellow oil is produced. The result is expressed, assuming 2.5 cubic centimeters of solution to have been used to produce the required result, as a color test of 97.5 (100 − 2.5 = 97.5).

The practical application of the color test may be made apparent by an example. A sample of a tank of 3,000 gallons of crude oil testing 1.5 per cent. in free fatty acids required 3.1 cubic centimeters of the caustic solution to produce a prime yellow color in the sample. The oil has then a color test of 96.9. The quantity of 20° Baumé caustic soda required for producing the standard color in the 3,000 gallons—in other words, to refine it—is ascertained thus:

$$3{,}000 \times .031 = 93 \text{ gallons}$$

This is the exact quantity of caustic soda at 20° Baumé necessary to refine the given quantity of oil.

When the strength of the solution is to be reduced, as is often necessary, sufficient water is added to make the total caustic solution equal to the desired bulk or percentage of the crude oil, as previously explained.

58. Determination of Free Oil in Cottonseed Foots. About 10 grams of foots is dried until free from all moisture (see *Quantitative Analysis*). To facilitate the operation of drying, a quantity of ignited white sand may be mixed with the foots. When it is also desired to ascertain the amount of moisture, the sand added must be accurately weighed and this weight deducted from the final weight of the dried foots. The thoroughly dried foots, with the sand, are transferred to a filter-paper capsule or thimble and extracted with light-boiling petroleum or rhigolene in the Soxhlet apparatus. The operation is carried out in the manner described later. The solvent is distilled off and the residual oil dried and weighed as there directed. After drying, the foots may also be extracted, as indicated later, and will then give an approximation sufficiently accurate for some technical purposes in the oil refinery or soap factory.

59. Determination of Total Fatty Acids in Cottonseed Foots.—As there is always more or less free oil in the foots, they must first be saponified. Any convenient weight may be taken, care being exercised first to mix and amalgamate thoroughly the oil and soapy matters to a homogeneous mixture. A convenient weight is 20 grams, as the foots usually contain a large percentage of moisture.

The known weight is transferred to a capacious porcelain or copper dish and about 1 ounce of strong caustic-soda solution placed on it; an addition of 25 or 30 cubic centimeters of methyl alcohol will materially aid in the rapid saponification of the oil while the mixture is being heated. The mixture is worked with the spatula while heating in the dish and the materials thoroughly incorporated and saponified at a low heat. The saponification will be accomplished

in from 10 to 15 minutes, as a rule. The crumbly soap is stirred with the spatula until all the alcohol has evaporated. The soap is tested for complete saponification by removing a portion and dissolving it in water. No permanent cloudiness or turbidity will be apparent when a thorough saponification has taken place. In the event of incompleteness in this respect, more caustic-soda solution and another addition of methyl alcohol is made to the material in the dish and the whole again heated and stirred. This second procedure, however, is seldom necessary.

When the mass in the dish has cooled, about a liter of hot water is added to it and the whole boiled until all lumps have been dissolved and a clear solution of soap is obtained. To this solution is then cautiously added dilute sulphuric acid in slight excess, when the fatty acids will separate and rise in clots to the top. The boiling of the contents of the dish is continued, with constant stirring to avoid loss by spurting, until a perfectly clear layer of the melted fatty acids appears on the surface.

The underlying acid water may be siphoned off from the fatty acids, care being taken, however, to avoid removing any of the fatty acids. The latter are washed several times with hot water and the siphoning repeated until the excess of sulphuric acid used has been thoroughly removed. The fatty acids are then transferred to a weighed dish, any adhering material being removed from the dish by light-boiling petroleum, or rhigolene, and added to those already in the weighed dish. The fatty acids are transferred to a drying oven and dried at 110° C. to a constant weight, and the percentage is calculated in the manner stated in the next article.

60. Instead of siphoning from the dish, application may be made of the following method, which, in very accurate operations, is to be preferred. After cooling somewhat, the contents of the dish is poured into a separating funnel of sufficient capacity and the underlying water withdrawn through the stop-cock. The dish that contained the fatty acids is now rinsed with the usual solvent and the washings

placed in the funnel with the rest of the fatty acids. The washing of the dish is continued until all traces of the fatty acids have been removed. The fatty acids are now washed in the funnel with hot distilled water until every trace of sulphuric acid has been removed from them by the successive washings. The washings should be saved for further treatment. The fatty acids are now drawn off into a tared beaker or capsule and dried, as usual, at 110° C. to constant weight. The separatory funnel must be rinsed in the same manner as the dish and the rinsings added to the fatty acids in the dish.

The wash water drawn off, if of too great bulk, is evaporated to a convenient quantity and shaken up several times with the petroleum ether or rhigolene to remove any fatty acids that may remain in them, which may be equal to ½ per cent. or more of the weight of substance taken. The solvent is withdrawn into either the receptacle containing the bulk of the fatty acids or it may be evaporated in another weighed dish and dried in the usual way. In the latter case, the two residual weights must be added to obtain the total weight of the fatty acids. From this weight the percentage of total fatty acids in the original foots can be readily calculated in the usual way by dividing the weight of substance obtained by the original weight of the sample taken and multiplying by 100 (see *Quantitative Analysis*).

61. Determination of Fatty Acids in Soap Stock. By means of the preceding method the fatty acids of soap stock may also be determined. In place of the preliminary saponification, the material may be dissolved at once in water and the operations followed as directed above. To be merchantable, cottonseed soap stock must contain at least 40, and in some cases 45, per cent. of fatty acids.

62. Cold Test for Cottonseed Oils.—Winter cottonseed oils are always sold subject to the cold test and must fulfil the requirements set forth in the rules of the New York Produce Exchange. The oil to be tested must be clear, and at the time of testing any stearin present must be com-

pletely dissolved in the oil. This test is accomplished by allowing the sample of oil to remain for 5 hours at a temperature of 32° F., at the expiration of which period the oil must appear clear, brilliant, and limpid.

It is at times desirable to ascertain the actual cold test of a cottonseed oil for certain specific uses. The method applicable in such cases is the following: A 4-ounce sample bottle is filled about half full of the oil to be tested and a thermometer passing through a cork, is extended well into the oil. The bottle thus arranged is placed in a freezing mixture and the oil frozen or congealed. The bottle is then withdrawn and, protected from the heat of the hand, the oil is stirred by the thermometer until it runs freely from one end of the bottle to the other. The thermometer is then withdrawn from the bottle and wiped with a piece of cotton waste sufficiently to ascertain the temperature. The reading may be taken as the actual cold test of the oil; in other words, the temperature at which, without reference to its clearness, the oil will remain limpid and flow.

63. Determination of Mealy Matter in Crude Oil. While merchantable crude oils as a rule are free from mealy material, which in reality is but fine cottonseed meal, some imperfectly settled or filtered oils contain appreciable amounts of it. The determination of mealy matter is a simple operation.

A known quantity of the crude oil is taken and filtered through a dry and previously weighed filter paper. The operation is materially shortened, as to the time required, by mixing the sample with two or three times its volume of carbon bisulphide. The filter paper retains the fine meal; the filter paper is then washed with successive portions of carbon bisulphide, dropping it around the top edge of the paper until the latter is washed thoroughly free from oil. The filter paper is dried as usual in the drying oven and then weighed with the adhering meal. The increase in weight is the amount of mealy matter present in the sample of crude oil taken. The percentage is found in the usual way.

Choice crude oil must be entirely free from this material. The usual care should be taken in sampling to secure a fair average and representative sample of the crude oil to be subjected to analysis.

64. Determination of Moisture in Cake or Meal. The cake is made into meal in the manner described later. Five grams of it is dried in the usual manner in the drying oven to a constant weight, which ordinarily requires about 3 hours at 105° C. The loss in weight is regarded as moisture (see *Quantitative Analysis*).

65. Determination of Oil in Cake or Meal.—In the average oil mill, an accurate analysis of the amount of cottonseed oil left in the pressed cake or ground meal is seldom made. Not that no analyses are made, but those which are made are more or less crude. They are sufficiently accurate, however, for working purposes in the mill, where strict accuracy is not as important as an immediate knowledge of the effective work that the presses are doing. Both the mill-working method of analysis and the method carried out by the analytical chemist will be described, as it is well to be familiar with both.

66. The *accurate determination* of oil in cake or meal is carried out as follows: The manner of taking an average sample influences to a remarkable extent the final result. This is especially true in the case of the oil cake, which must be reduced to a fine powder before weighing. The cake selected for the test is sawed through diagonally both ways and equal portions are again sawed from each of these pieces, care being taken to have as representative a sample as possible from all parts of the cake—the center, middle portion, and the edge. The meal dust and selected pieces may be separated by passing through an 80-mesh sieve. The fragments in the sieve are reduced to fine powder in a porcelain mortar and again screened, and this procedure is followed until all the portion of the cake selected for analysis has been passed through the screen. The sample is then

thoroughly and intimately mixed, as usual with material of this nature, to obtain an average sample. Five grams of the meal, or any definite weight, as 20 grams, is accurately weighed off in a small porcelain dish and dried to a constant weight, as in the determination of moisture, previously given.

The dried meal is then transferred into a folded filter paper or a filter-paper capsule (the latter being preferable), and placed in the Soxhlet extraction apparatus. The solvent used may be ether, light-boiling petroleum, or carbon bisulphide. The latter is in general use for this determination. The general arrangement of the apparatus may be seen in Fig. 5, in which a tared flask containing the solvent is shown at *b*; the Soxhlet extraction apparatus at *a*; the condenser cooled by appropriate water supply at *d*; and the filter-paper capsule containing the cottonseed meal from which the oil is to be extracted, at *e*. The meal in the capsule *e* is kept from being carried over with the solvent by a wad of absorbent cotton placed on top of it. The capsule containing the meal having been placed in the tube, the solvent is placed in the flask, the weight of which is accurately known, and the heat applied to boil the solvent. As the solvent boils, the vapor is condensed and falls directly into the capsule on the meal, remaining in contact with it until the solvent is siphoned off by the attachment *c* on the apparatus. The solution passes down into the flask to be again boiled and recondensed, this operation being continued until all the oil has been extracted, which usually requires about 1 hour if the extraction is regular. By properly arranging the source of heat, the operation is performed regularly and automatically.

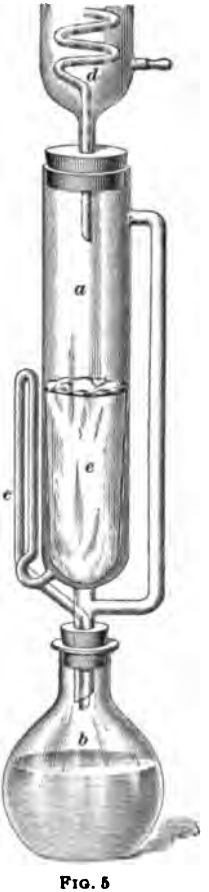


FIG. 5

As all the solvents just mentioned are very inflammable, great care must be taken, especially when heating by the naked flame, to have the corks of the apparatus air-tight and to have a perfect condensation of the solvent. As carbon bisulphide has a very low boiling point, it is far preferable in the oil mill to have steam or hot water as the source of heat, thus guarding against undue risk of fire by possible accidents through breakage.

When the extraction is complete, the Soxhlet apparatus is disconnected from the condenser *d* and the thimble *e* is removed. The apparatus is then set up as before and the volatile solvent distilled into the space that the thimble with the contained meal occupied in the process of extracting the oil. When the solvent has nearly reached the level of the top of the tube *c*, the apparatus is again disconnected and the distillate emptied into a convenient bottle. This operation is continued until the greater portion of the solvent used in the extraction of the oil has been recovered. The redistilled material may thus be saved and utilized for further extractions. The residue in the flask is then placed in the drying oven at a temperature of 105° C. to drive off the last traces of the solvent from the extracted oil. From 2 to 3 hours, depending on the quantity taken for analysis, is sufficient for this operation. The usual calculation, dividing the residual weight by the weight of substance taken and multiplying by 100, gives the percentage of cottonseed oil in the cake or meal. In the analysis of cottonseed meal, as the material is already ground, it is only necessary to have it of the fineness mentioned and to insure an average representative sample being taken.

67. The *approximate determination* of oil in cake or meal is fairly accurate and well adapted for working in the oil mill, where rapid results closely approaching the truth are required.

The sample is prepared in the same manner as has been described. A known weight of meal is placed in a tube *a*, Fig. 6, about 10 inches long and $1\frac{1}{2}$ inches in diameter, the

lower part being drawn out to a tube about 2 inches long and ¼ inch in diameter. The material *b* is packed fairly tight on a wad of absorbent cotton *c* in the bottom of the large tube, and another wad of cotton is placed on top of the material. The latter plug is serviceable for removing any fine particles of meal remaining on the weighing glass. A weighed porcelain dish having been placed underneath the tube, the latter, supported in an upright position, is filled with carbon bisulphide. The solvent in slowly percolating through the meal readily dissolves the oil, the solution dropping into the weighed porcelain dish beneath. After the first addition of the solvent has gone through the meal, successive additions in the tube are made until the carbon bisulphide comes through colorless, when all the oil has been extracted. As the solvent evaporates very rapidly, a small-sized dish is sufficient to contain the solution of oil.

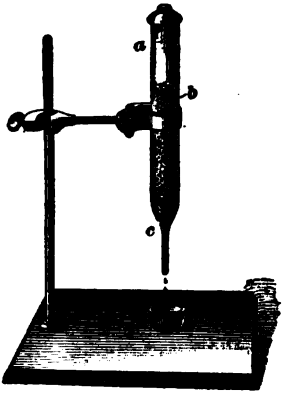


FIG. 6

When all the oil has been dissolved out, the end of the tube is washed with a small quantity of the solvent to remove any adhering oil. The solution of oil in the dish has the carbon bisulphide driven off and is then dried in the oven at 105° C. to a constant weight. The usual calculation gives the percentage of oil that was in the cake or meal. During the operation, the tube is kept covered with a suitable porcelain crucible cover to prevent a rapid evaporation of the solvent. Extreme care must be taken to have no fire or flame in the vicinity where this operation is being carried out.

The dimensions of the tube may be varied to suit individual convenience; in some mills, a tube 15 inches long and ½ inch in diameter is used.

ANALYSIS OF COTTONSEED FOR YIELDS OF OIL, KERNELS, HULLS, AND CAKE OR MEAL PER TON OF SEED

68. It is sometimes very desirable to ascertain what a certain lot of cottonseed will yield in mill working. The following methods are usually employed for ascertaining the probable yields of the different products, and they give, when a fair average of the seed is taken, results very close to the truth.

The sample of seed is spread on a large sheet of paper and mixed. Ten or fifteen seeds are taken from this sample and split through lengthwise with a sharp knife. The kernels are put on one watch glass and the hulls on another. The hulls and kernels are weighed separately and the two weights added together. Thus, for example:

	GRAMS
Weight of kernels	1.100
Weight of hulls	.790
Weight of hulls and kernels, or weight of seed taken	1.890

The weight of the kernels is divided by the weight of the seed taken, or $1.100 \div 1.890 = .582$, and this figure multiplied by 100 for percentage gives 58.2 per cent. or, in other words, 58.2 pounds of kernels to each 100 pounds of seed. In the same manner, the weight of the hulls is divided by the weight of the seed, or $.790 \div 1.890 = .418$, which, multiplied by 100 for percentage, gives 41.8 per cent., or 41.8 pounds of hulls per 100 pounds of seed.

The kernels that have been previously weighed are now crushed to a fine powder in a mortar in order to break up all the oil cells in the meats. From this powdered kernel meal is weighed out exactly 1.100 grams of the meats. As it is impossible to remove from the mortar all the powdered meat, two or three more kernels should be added to the mortar before the material is crushed, in order to obtain the proper quantity for making the required weight.

The weighed-out quantity of crushed kernels is extracted for oil in the usual manner, as given under the head of

Analytical Methods. After the oil is extracted, it is weighed as usual. Assume the weight of oil obtained to be .450 gram. Dividing this weight of extracted oil by the weight of the kernel and meal taken gives .450 ÷ 1.100 = .409; multiplying this by 100, as usual, for percentage, gives 40.9 per cent., or 40.9 pounds of oil in each 100 pounds of meat. The meal in the extracting tube or Soxhlet apparatus will weigh .65 gram, this being the difference between the weight of the original meats taken and the oil extracted. Dividing this weight by 1.100, the quotient will be .591, and this, multiplied by 100, gives 59.1 per cent., or 59.1 pounds of meal in each 100 pounds of meat. This is the percentage of cottonseed meal absolutely free from oil. By subtracting the percentage of oil found by this method from 100, the same result may be obtained without the latter calculation.

From the foregoing, it is found that cottonseed containing 58.2 per cent. of kernels, or meats, yields 40.9 per cent. of oil and 59.1 per cent. of meal.

69. Determination of the Percentage and Pounds of Oil per Ton of Seed.—In the preceding determination, it is seen that the kernels from the seed contain 40.9 per cent. of oil. By multiplying the percentage of kernels found in the whole seed by the percentage of the oil, or 58.2 × 40.9, it will be found that 23.8 per cent. of oil is contained in the whole seed. As it is impossible in practical working to extract all the oil from the meats by pressure, a fair average loss estimated at 7 per cent. left in the cake is a convenient factor for calculation. By subtracting 7 per cent., which is assumed will be left in the cake in good economical working, from 23.8 per cent., which is the amount of oil contained in the whole seed, the available oil that should be extracted from the seed is found to be 16.80 per cent., which is equivalent to 16.80 pounds of oil in every 100 pounds of whole seed. Multiplying this result by 20, the number of hundredweights to the ton, gives as a result 336 pounds, or the amount of oil to be extracted from each ton of seed in the mill. As there are 7.5 pounds of oil in each gallon, to

obtain the number of gallons of oil per ton of seed, as it is usually stated, the 336 pounds is divided by 7.5, giving as a result 44.8 gallons of oil per ton of seed.

70. Determination of the Percentage and Pounds of Meal per Ton of Seed.—From the foregoing, it is seen that the kernels of the seed yielded 59.1 per cent. of meal. Multiplying the percentage of the kernel found in the whole seed by the percentage of meal, or 58.2×59.1, gives 34.4 per cent.; or, in other words, there are 34.4 pounds of meal in each 100 pounds of whole seed. As there are 20 hundredweights to each ton, multiplying 34.4 by 20 gives as a result 688 pounds of meal. As, however, there has been deducted 7 per cent. or 7 pounds of oil from the oil determination, this amount being retained in the commercial cake as fat or oil, the same correction must be applied to the ton results; in other words, there will be retained in every 20 hundredweights, or ton, of whole seed, 140 pounds of oil, and this must be added to the cake or the meal. Adding 140 pounds to 688 pounds gives 828 pounds, which is the quantity of meal that is obtained from each ton of seed.

71. Determination of the Percentage and Pounds of Hulls per Ton of Seed.—The whole seed, as just noted, was found to contain 41.8 per cent., or 41.8 pounds of hulls to each 100 pounds of whole seed. Multiplying by 20 to obtain the ton percentage gives 836 pounds, or the number of pounds of hulls per ton of seed.

72. Determination of Meats or Kernels in Mill Hulls.—In testing hulls for meats, the sample is quartered in the usual way until from 4 to 8 grams remain. This sample is washed with ether in a tube (Fig. 6) or may be extracted in the ordinary extraction apparatus. For convenience, the meats left in the hulls are regarded as a multiple of the oil, and while the proportion varies with different seeds, it is sufficiently accurate to take one-third, or $33\frac{1}{3}$ per cent., as the multiple. If, for example, the hulls are shown to have 1.78 per cent. of oil, yielded by the extraction, it is assumed that they contain three times as much

meats, or 5.34 per cent., which is equivalent to 5.34 pounds in every 100 pounds of hulls, or to 106.8 pounds in every ton of hulls. It is sufficiently close in mill-working analysis to consider the hulls as 45 per cent. of the whole seed, and, multiplying the 106.8 pounds of meats in the hulls by this percentage, gives, as a result, 48.06 pounds, or the amount of meats left in the hulls from working 1 ton of seed.

One-quarter of 1 per cent. of oil obtained from the hulls by the preceding method is considered as low a percentage as it is practicable to obtain in ordinary mill working.

73. Determination of Fertilizer Constituents. The usual estimations for nitrogen, phosphoric acid, and potash, described in *Quantitative Analysis*, are applied to cottonseed cake and meal. For the estimation of nitrogen, the Kjeldahl method should be employed, as it is the most convenient (see *Quantitative Analysis*).

The following modification of the Kjeldahl apparatus has been found very serviceable. In place of the usual simple bent tube extending from inside the flask *A* into the condenser *d*, the bulb *a*, as shown in Fig. 7, is employed. The tube *b* is blown in the bulb *a*, its shape allowing of rapid and violent boiling without incurring the risk of having any particles of the alkaline solution projected and carried over into the distillate of ammonia. The bulb is drawn out to a tube end *c*, which passes through a cork into the flask *A*.

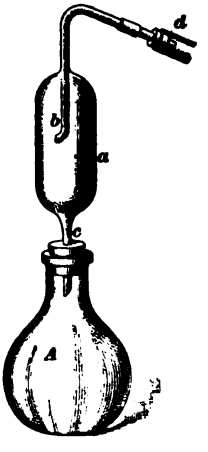


FIG. 7

An average analysis from many samples of cottonseed meal made from decorticated seed showed the following percentages of fertilizer constituents: Nitrogen, 7.25 per cent. (equivalent to ammonia 8.80 per cent.); phosphoric acid, 2.24 per cent.; and potash, 1.74 per cent. These percentages will vary, naturally, with the amount of moisture present in the meal and with the quality of the seed originally employed.

ANALYSIS OF MEAL OR CAKE FOR FEED-STUFF CONSTITUENTS

74. Determination of Protein.—The nitrogen in the substance is determined by the Kjeldahl method in the usual way and the percentage of nitrogen is multiplied by 6.25, the factor employed for the purpose of determining the protein content. The nitrogen may be determined by any desired method, but the Kjeldahl method has been found most convenient.

75. Estimation of Crude Fiber.—Two grams of the substance is extracted with ordinary ether, or the residue from the ether-extract determination may be used for the estimation of crude fiber. To this residue, in a 500-cubic-centimeter flask, add 200 cubic centimeters of boiling sulphuric acid of 1.25 per cent.; connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once and continue the boiling for 30 minutes. A blast of air conducted into the flask serves to reduce the frothing of the liquid therein. After the required period of boiling, the liquid is filtered and washed with boiling water until the washings are no longer acid. The substance on the filter is rinsed back into the same flask with 200 cubic centimeters of a boiling 1.25-per-cent. solution of caustic soda, free, or nearly so, from carbonate of soda. This solution is at once boiled again for 30 minutes in the same manner as for the treatment with acid. Filter on a Gooch crucible and wash the substance until the washings are neutral. The crucible and contents are dried at 110° C. and the weight noted. The substance on the crucible is completely incinerated and the crucible cooled and again weighed. The loss in weight represents the crude fiber in the material.

The filter used for the first filtration may be linen or any other kind that secures clear and reasonably rapid filtration. The solutions of sulphuric acid and caustic soda are to be made up of the specified strength, which should be deter-

mined accurately by titration and not merely from specific gravity.

76. Determination of Ash.—Char from 2 to 3 grams of the substance in a platinum crucible and burn to whiteness at the lowest possible red heat. For this purpose the ordinary muffle furnace is very convenient. If a white ash cannot be obtained in this manner, exhaust the charred mass with water and collect the insoluble residue on a filter. This residue is burned and the ash added to the residue from the evaporation of the aqueous extract. The whole is then heated to a low redness until the ash is white or nearly so.

77. Determination of Ether Extract.—To prepare the anhydrous alcohol-free ether required for the estimation of fat, any one of the commercial brands of ether is washed with two or three successive portions of distilled water and solid sticks of caustic potash or soda are added until most of the water has been extracted from the ether. Carefully cleaned metallic sodium is cut into small pieces and added until there is no further evolution of hydrogen gas. The ether thus dehydrated must be kept over metallic sodium, and should be only lightly stoppered in order to allow any accumulating hydrogen gas to escape. The ether may be drawn off with a pipette as required.

In the determination of ether extract by the direct method, from 2 to 3 grams of the substance, after drying, is extracted with anhydrous alcohol-free ether for 16 hours. The extract thus obtained is dried as usual to a constant weight. This is termed the ether extract, which is sometimes designated as crude fat.

In the indirect method, the moisture is determined as described in the following article and the dried substance extracted in the Soxhlet apparatus for 16 hours. The substance is again dried and the loss of weight regarded as ether extract, or crude fat.

78. Determination of Moisture in Feeds.—To determine the moisture in feed stuffs, the substance is ground and passed through a sieve with circular holes 1 millimeter in

diameter. The sample prepared in this manner is used in all the determinations for feeding stuffs. From 2 to 3 grams of the material is dried for 5 hours, at a temperature of boiling water, in a current of hydrogen or in vacuo. If the substance is contained in a glass vessel, the latter must be kept from contact with the boiling water. The loss in weight will be the moisture contained in the substance.

79. Determination of Carbohydrates.—The carbohydrates are determined as (*a*) the reducing sugars (estimated as dextrose), (*b*) the sucrose, and (*c*) the starch (see *Quantitative Analysis*, where the methods for the determination of sugars are given in full). In the analysis of cattle feeds, 3 grams of the prepared sample is stirred in a beaker with 50 cubic centimeters of water for 1 hour. The solution is then filtered into a 250-cubic-centimeter flask, washed, and the solution made up to the 250-cubic-centimeter mark.

(*a*) The *sugar* is determined as dextrose, according to the methods described in *Quantitative Analysis*. If the solution is difficult to filter clear, 2 cubic centimeters of alumina cream should be added.

(*b*) The *sucrose* is determined in 50 cubic centimeters of the filtrate obtained above in the manner described in *Quantitative Analysis*. Before calculating the invert sugar obtained as sucrose, the reducing sugar obtained as dextrose must be deducted.

(*c*) The *starch* is determined in the insoluble residue from the filtration of the sample of 3 grams taken. The residue is heated for $2\frac{1}{2}$ hours with 200 cubic centimeters of water and 20 cubic centimeters of hydrochloric acid (1.125 specific gravity) in a flask provided with a reflux condenser. The contents of the flask is then cooled and neutralized with sodium carbonate. The volume of solution is made up to 250 cubic centimeters, filtered, and the dextrose determined in an aliquot portion of the filtrate. The weight of dextrose obtained multiplied by .9 gives the weight of the starch. In this method, there will be included as starch the pentoses and other carbohydrate bodies present, which suffer hydrolysis

and conversion into reducing sugars on boiling with hydrochloric acid.

(*d*) Starch may also be estimated by the following method, which has recently been devised; it gives good results in the determination of this carbohydrate. According to L. Gianturco, the aluminum hydrate thrown down from an aluminum-salt solution and dried at 100° C. corresponds with the formula $Al(OH)_3$. When calcined in a platinum crucible, it changes into Al_2O_3. If starch is suspended in water, and to this mixture is added a definite quantity of a standardized solution of an aluminum salt, and the aluminum is precipitated as hydrate by means of ammonia, the starch is carried along by the aluminum hydrate, the precipitate being gathered on a filter and washed with as little water as possible until the wash water contains no more sulphates. If this mixture is dried and weighed, the difference between the weight found and the known weight of the aluminum hydrate, corresponding to the used volume of alum solution, furnishes the weight of the starch. Finally, the dried deposit may be calcined in a platinum crucible to a constant weight, whereby the aluminum oxide Al_2O_3 + ash of the starch remains as residue. If by this method a considerably larger amount of aluminum oxide should be found than can be computed from the employed amount of the titrated alum solution, the presence of a considerable quantity of insoluble mineral substances would be proved in the starch. The alum solution employed by L. Gianturco contained .060769 gram of the salt per cubic centimeter, corresponding with .01 gram of aluminum hydrate. It is claimed that this method gives better results than any other estimation method.

80. Rapid Method of Estimating the Iodine Number in Oils, Greases, Etc.—The following recently devised modified method, originally carried out with greases and lard, has been found very serviceable and reliable for oils. For a quick determination of the iodine number in greases, J. Ballier uses a solution of 50 grams of iodine and 32 grams of bromine in glacial acetic acid, or a solution of 33.5 grams

of iodine and 42.2 grams of bromine in the same solvent, saturating the liquid, which amounts to 950 cubic centimeters, with mercury bichloride (about 95 grams being required). The solution is ready for use in 2 days, and is so made by diluting with glacial acetic acid that 1 liter of it corresponds to 100 grams of iodine; that is, 5 cubic centimeters of the bromine-iodine solution requires 39.4 cubic centimeters of $\frac{n}{10}$ thiosulphate solution for discoloration. As a solvent for the greases, a mixture of 500 cubic centimeters each of glacial acetic acid and chloroform is used. This mixture is saturated with mercury bichloride, and 10 cubic centimeters of a 10-per-cent. solution of potassium iodide and as much bromine-iodine solution as is required for the liquid to remain colored a yellowish red, are added.

In making the determination of the iodine number, dissolve 1 gram of grease or oil in 20 cubic centimeters of the solvent mixture until the resulting yellowish-red tint remains constant for at least 5 minutes. The results are said to coincide approximately with those of the Von Hübl method. The computation is as follows: Since 1 gram of fat or oil is used and the bromine-iodine solution contains exactly 10 per cent. of iodine, or an equivalent quantity of bromine, the iodine value is found by multiplying the number of cubic centimeters of the bromine-iodine solution used by 10. In order that the standard of the bromine-iodine solution shall remain constant, it is well to charge the burette by means of a pipette, and not by direct pouring in.

For the usual method of determining the iodine number, reference should be made to *Quantitative Analysis.*

81. Detection of Cottonseed Oil in Mixtures.— Among the most recent contributions to the scientific investigation of cottonseed oil is that of Raikow, who has demonstrated that this oil contains organic chlorated compounds. Upon this characteristic, which is not possessed by olive oil or nut oils, is based a new method for the identification of cottonseed oil in mixtures.

(*a*) The *presence of chlorine* in cottonseed oil may be detected by burning it and collecting the products of combustion in a solution of caustic potash free from all traces of chlorine and sulphur. The solution is then acidulated with pure nitric acid, and precipitation of the chlorine present is effected with nitrate of silver.

(*b*) The *presence of sulphates* may be detected by boiling the solution obtained with the combustion products, and, after acidulating it with hydrochloric acid, adding the solution of barium chloride in the usual manner. Although it has been claimed by some that cottonseed oil contains volatile sulphureted compounds, the most recent investigations on oils from many different localities have failed to confirm this claim. The organic chlorated compounds present in all cottonseed oils are not soluble in water and only slightly soluble in alcohol.

Where the amount of cottonseed oil mixed with olive or nut oils is small in quantity, a larger amount of the mixed oil must be taken for analysis. In such a case, about 4 or 5 grams is burned and the products of combustion collected by aspiration and passage through a wash bottle containing potash solution (see *Quantitative Analysis* for the adaptation of the apparatus necessary for this procedure). In the average mixture of these oils, 1 gram is sufficient for analysis. The entire absence of chlorine compounds in olive and nut oils and the presence of these compounds in cottonseed oil gives to this test a positiveness that has heretofore been lacking in the various methods of examining mixtures of olive oil and cottonseed oil where the latter is present only in small quantities.

The quantity of chlorine in different cottonseed oils varies largely, and up to the present time it has not been definitely ascertained whether these chlorine compounds exist in the oil before or after its preparation.

82. Halpen Reaction for Cottonseed Oil.—Another method for the detection of cottonseed oil in mixtures that gives very reliable results is known as the **Halpen reaction**

test. This test is more sensitive than the Bechi test (see *Quantitative Analysis*) and less liable to give unsatisfactory results in the hands of an inexperienced person. It is not affected by rancidity, which circumstance is of great importance at times. The depth of color is proportional to a certain extent to the amount of oil present, and by making comparative tests with cottonseed-oil mixtures of known proportions, some idea as to the amount of cottonseed oil present in a mixture of oils under examination can be obtained. It must be remembered, however, that different oils react with different intensities and cottonseed oils that have been heated to 200° to 210° C. react with greatly diminished intensity. Heating 10 minutes at 250° C. renders cottonseed oil incapable of giving reaction.

Lard and lard oil from animals fed on cottonseed meal will give a faint reaction, as will also the fatty acids.

The Halpen test is made as follows: Carbon bisulphide containing about 1 per cent. of sulphur in solution is mixed with an equal volume of amyl alcohol. Equal volumes of this reagent and the oil under examination are mixed and then heated in a bath of boiling saturated brine for from 1 to 2 hours. In the presence of as small an amount as 1 per cent. of cottonseed oil, a characteristic orange or red color is produced.

In the Bechi, or silver-nitrate, test for cottonseed oil, it should be noted that rancid oils or fats must be purified before testing. This requirement, however, does not have to be considered in Halpen's test.

PETROLEUM AND PRODUCTS

(PART 1)

NATURE AND COMPOSITION OF PETROLEUM

1. Petroleum, also known as rock oil, maltha, and Seneca oil, is one of nature's products that is widely divided over the surface of the earth. It has been found in springs, oozing from the ground, in shallow wells, and Artesian wells many thousand feet deep. In this country, it is found in one or the other of its various forms in almost every state, although it seems to occur in paying quantities in comparatively few of them.

It is found in Pennsylvania, New York, and West Virginia, in sandstone of various geological formations, which is found in several distinct layers, known as the first, second, third, fourth, and fifth oil sands. We thus find wells of various depths; while a well, say 60 or 70 feet deep, may be drilled to the first sand and produce oil, another, perhaps, must be drilled to the third or fourth sand before oil is found and may be 600 feet or more deep.

2. Chemical Composition of Petroleum.—Petroleum is composed in varying quantities of almost the entire series of pure hydrocarbons known. In American petroleum, the methane or C_nH_{2n+2} series seem to predominate. Sadtler found natural gas, which is now generally acknowledged to belong to the same class and have the same origin as petroleum, to be composed almost entirely of methane, ethane,

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and propane. The first, or marsh gas, constituted about three-fourths of all the gas analyzed. Peckham gives the following analysis of a number of noted American gas wells.

Well	CO_2	CO	H	CH_4	C_2H_6	C_3H_8
Burns.....	.34	trace	6.10	75.44	18.12	trace
Leechburg.....	.35	.26	.56	89.65	4.39	trace
Harvey.....	.66	trace	13.50	80.11	5.72	trace

Burns's well is in the northern part of Butler County, Pennsylvania; Leechburg is in Armstrong County, and the Harvey well is in the southern part of Butler County. Nitrogen has been found in the gas from the Baku fields, but very little has been reported to exist in any of the Pennsylvania gas.

3. The first chemical examination of the liquid products of the wells, that seems to concern the petroleum industry, was that of Professor Benjamin Silliman, Jr., in 1855. In the earlier period of the industry it was assumed that petroleums from different localities were identical, except in specific gravity and that, therefore, the distillate of the same specific gravity possessed the same properties. Chemical analysis has shown that this by no means is the case.

4. While the methane, or marsh gas, series predominates in America, the naphthene, or more condensed series, predominates in Russia. This gives rise to a marked difference in the general properties of the products obtained from these crude oils. Mendeleeff considers that although the two crude oils contain almost the same hydrocarbons, the proportion is far from being the same. From a large number of Baku crude oils examined by him, he found that the specific gravity of the distillate, obtained between 100° and 105° C., after four or five fractionations varied between .701 and .756; while the corresponding fraction of American petroleum lies between .703 and .71.

TABLE I

HYDROCARBONS, C_nH_{2n+2}, ISOLATED FROM AMERICAN CRUDE OIL

Name	Chemical Formulas	C	H	Boiling Point	Specific Gravity	Remarks
GASEOUS						
Methane	CH_4	75.00	25.00		.559	
Ethane	C_2H_6	80.00	20.00		.5516	
Propane	C_3H_8	81.81	18.19	−20° C.	1.522	Colorless liquid at −20° C.
Butane	C_4H_{10}	82.80	17.20	1°	.600 (0° C.)	Colorless liquid of sweet taste
LIQUID						
Pentane	C_5H_{12}	83.33	16.67	37°	.628	
Hexane	C_6H_{14}	83.72	16.28	69°	.664	
Heptane	C_7H_{16}	84.00	16.00	97.5°	.699	
Octane	C_8H_{18}	84.21	15.79	125°	.703	
Nonane	C_9H_{20}	84.38	15.62	136°	.741	
Decane	$C_{10}H_{22}$	84.51	15.49	158°	.757	Flash 26° C., fire 38° C.
Undecane	$C_{11}H_{24}$	84.61	15.39	182°	.765	Flash 40° C., fire 58° C.
Dodecane	$C_{12}H_{26}$	84.70	15.30	198°	.776	
Tridecane	$C_{13}H_{28}$	84.78	15.22	216°	.792	
Tetradecane	$C_{14}H_{30}$	84.85	15.15	238°	.812	
Pentadecane	$C_{15}H_{32}$	84.90	15.10	258°	.825	
Hexadecane	$C_{16}H_{34}$	84.94	15.06	280°	.828	
SOLID						
Paraffin myricyl	$C_{31}H_{66}$	85.26	14.74			
Paraffin ceryl	$C_{30}H_{62}$	85.31	14.69	370°		

5. We are indebted to Schorlemmer, Pelouze, Cahours, and Warren for our knowledge of the composition of petroleum. The compounds of the methane series given in Table I have been isolated from Pennsylvania petroleum and examined.

6. Besides the methane series, given in Table I, isomers of a great many of these hydrocarbons, having the same composition and specific gravity but a slightly different boiling point, have been isolated and identified. Höfer has isolated a series of olefines; Chandler has found benzene and some of its homologues in the Bradford crude; and Peckham gives an extended account of the discovery of petrocene and thallene in the tar distillation of petroleum. A number of other compounds have been mentioned as existing in small quantities; but since it is impossible to find many of these compounds without distilling large quantities of the crude oil, it is quite likely that many of them are simply products of condensation caused by the heat of the still.

7. The relative proportions of these different compounds existing in petroleum have never been fully determined. Yet an approximate idea may be obtained by distilling Pennsylvania petroleum and referring the results to Tables I and II. The latter table gives the commercial products and the compounds supposed to be contained therein.

8. Oxygen is shown to exist in nearly all petroleum, but much of this may be due to absorption, since all distillates are known to absorb it quite freely. Nearly all the oxygen is contained in the form of acids and phenylated compounds.

Table III, which was prepared by Aisinman, gives the elementary composition of petroleum from various localities.

9. Besides the three elements given in Table III, nitrogen has been found in nearly all crude oils. This element is usually present in the form of bases. Peckham gives it as his opinion that it exists not only as pyridine and quinoline, but that a large number, if not all, of the methylated compounds of these bases are associated with them.

TABLE II

PRODUCTS MANUFACTURED FROM PETROLEUM

Name	Boiling Point. Degrees Centi-grade	Specific Gravity	Use
Natural gas	Gas		Fuel
Rhigolene	0°	.6222	Local anæsthetic
88° Gasoline	50°	.6400	Extraction of oils and air-gas machines
80° Gasoline	70°	.6660	Air-gas machines
Stove gasoline	98°	.7000	Fuel in a special stove
63° Deodorized	110°	.7250	Substitute for turpentine
Kerosene	150°	.7860	Illuminant
Lubricating oils		.8750—.9150	Lubricants
Vaseline		.8917—.9333	As a vehicle for salves
Paraffin			Candles and insulation

10. The element that has been of especial annoyance to the American refiner is sulphur. This is found in Ohio, Indiana, and Canadian oils in such quantities as to give them a marked odor that increases during distillation. Mabery has made an extended examination of these oils, and reports the percentage of sulphur in the Ohio and Indiana crudes never to exceed .6 per cent. He states that these compounds are sulphides of methyl, ethyl, normal propyl, normal and isobutyl, pentyl, ethyl pentyl, butyl pentyl, and hexyl, but that neither mercaptan nor thiophene were found. In Canadian petroleum he found .98 per cent. of sulphur, besides a quantity present as hydrogen sulphide.

11. Inorganic substances are always found in petroleum, but only in minute quantities. Silver, copper, mercury, iron, arsenic, and even gold have been found in the ash of

TABLE III

Locality	Sp. Gr. at 0° C.	Elementary Composition			Remarks
		C	*H*	*O* by Difference	
Baku	.882	87.40	12.60	.10	It is composed of naphthenes and a few other hydrocarbons, as well as oxygen and sulphur compounds; in the lighter fractions small quantities of paraffin are found.
Baku	.884	86.30	13.60	.10	
Baku	.897	86.50	12.00	1.50	
Baku	.928	87.10	11.70	1.20	
Baku, very heavy	.938	86.60	12.30	1.10	
Baku	.954	85.30	11.60	3.10	
Baku		86.00	13.00	2.00	
Pennsylvania, near Franklin	.886	84.90	13.70	1.40	The Pennsylvania crude oil consists principally of the paraffins, with a small percentage of the aromatic series and a minute quantity of the naphthenes in the heavy distillates. All American petroleum contains oxygen and sulphur compounds and a few, also, nitrogen compounds.
Oil Creek	.816	82.00	14.80	3.20	
W. Canada	.857	81.30	13.40	2.30	
Petrolia	.870	84.50	13.50	2.00	
West Virginia	.841	84.30	14.10	1.60	
West Virginia	.857	83.20	13.20	3.60	
West Virginia	.897	83.60	12.90	3.50	
Kentucky		85.20	13.36	1.11	
California		86.93	11.82	2.70	
California		86.62	12.92	.54	
Ohio	.887	84.20	13.10	.70	
Ohio, Mecca		86.30	13.07		
West Galicia	.855	85.30	12.60	2.10	A mixture of paraffins, naphthenes, and aromatic hydrocarbons.
East Galicia	.870	82.20	12.10	5.70	

TABLE III—*Continued*

Locality	Sp. Gr. at 0° C.	Elementary Composition			Compounds Found in Petroleum
		C	*H*	*O* by Difference	
Roumania	.901	83.00	12.20	4.80	
Roumania	.770	82.60	12.50	4.90	
Italy, Parma	.786	84.00	13.40	1.80	
Italy, Piedmont	.919	86.40	12.20	1.40	
Germany, Hanover	.892	80.40	12.70	6.90	Principally paraffins, contains also aromatic hydrocarbons and naphthenes.
Germany, Oberg	.944	84.40	11.50	4.10	
Germany, Witze	.955	86.20	11.40	2.40	
Germany, Alsace	.912	86.90	11.80	1.30	
Germany, Pechelbronn	.968	85.60	9.60	4.60	
Germany, Pechelbronn	.892	85.70	12.00	2.30	
Germany, Pechelbronn		86.10	12.70	1.20	
Germany, Pechelbronn		83.30	11.10	1.10	
Germany, Schwabweiler	.861	86.20	13.30	.50	
Germany, Schwabweiler	.829	79.50	13.60	6.90	
Germany, Schwabweiler	.820	85.50	14.20	.30	
East India, Burmah	.875	83.80	12.70	3.50	
Java, Rembang	.923	87.10	12.00	.90	
Java, Tjabados, Fanga	.827	83.60	14.00	2.40	
Java, Gagor	.927	85.00	11.20	2.80	
China, Fu-li-fu	.860	83.50	12.90	3.60	

petroleum. These elements seem to have but little effect on the technology of petroleum, which is affected more by the varying quantities of light and heavy hydrocarbon compounds that exist in petroleum than by almost any other factor in the composition.

TECHNO-CHEMICAL EXAMINATION

12. This examination is of much greater importance to the manufacturer than any ultimate analysis. Under this head will first be considered such physical properties as color and specific gravity. The color of the American crude petroleum varies from a clear water-white, through a straw-yellow, light amber, red, deep red, dark brown, to an opaque black.

The first, although only a freak in the petroleum fields, has been found in a well at Holders Run, Armstrong County, Pennsylvania. The others are of common occurrence and may be seen in the crude oils of West Virginia, Pennsylvania, Ohio, Indiana, California, and Wyoming, respectively. Each of these States has produced crude oils of various shades, the light-colored oils usually coming from some strata nearer the surface than those of a darker shade. Table IV, taken from Aisinman's Taschenbuch, gives these physical properties for petroleums from several localities.

13. Nearly all crude oils have a fluorescence varying from blue to dark green. That of Pennsylvania is of a beautiful light grass green. The specific gravity of crude oil varies from .77 to .98, all being lighter than water. By referring to Table IV, it will be noticed that there exists a certain connection between color and specific gravity, the lighter colored oils usually having a lighter specific gravity, and vice versa.

14. The variability mentioned in the preceding articles depends partly on the chemical composition and partly on the physical condition of the oil. All crude oils hold more

or less natural gas in solution, which lowers their specific gravity; on the other hand, oils containing a comparatively large percentage of paraffin or asphalt have a greater specific gravity—this also applies to all sulphur oil. It has been further found that oils having a low specific gravity furnish

TABLE IV

COMPARISON OF COLOR AND SPECIFIC GRAVITY OF CRUDE OILS

Specific Gravity	Color	Odor	Locality
.777	Light straw	Strong, unpleasant	Persia
.787	Light straw	Pleasant	Near Milan, Italy
.810	Light amber	Pleasant	Bradford, Pennsylvania
.818	Light amber	Faint, pleasant	Mud volcano, Burmah
.828	Light brown	Very faint	New Zealand
.829	Amber	Unpleasant	India
.835	Dark brown	Faint, pleasant	East Borongo
.836	Dark reddish brown	Faint, pleasant	Russia
.843	Dark brown	Faint, pleasant	Hanover
.852	Dark reddish brown	Faint	South America
.865	Dark brown	Penetrating	Canada
.866	Dark brown	Faint, pleasant	Burmah
.888	Chestnut brown	Faint, pleasant	Burmah
.900	Dark brown	Very faint, somewhat viscous	South America
.910	Black	Unpleasant	Wyoming
.913	Brown-black	Unpleasant	Hanover
.933	Dark brown	Faint, pleasant, viscous	Assam
.935	Dark brown	Viscous	India
.942	Brown-black	Viscous	Russia
.945	Black	Unpleasant	Wyoming
.957	Black	Tar	Barbadoes, C. A.

a greater percentage of gasolines and illuminating oils than those that are heavier; the specific gravity of an oil plays an important part in the price of the oil.

The more important factor in setting the price of oil, however, is its chemical composition and freedom from

suspended matter. Heavy oils hold water and earthy matter in suspension and must be heated in order to settle them before they are received by the pipe line. This causes a loss of the more volatile parts and necessarily makes them less valuable. The sulphur oils, besides being heavier, require a more expensive treatment in order to produce a first-class illuminating oil and, consequently, are not so desirable to the refiner.

When an oil contains very small percentages of gasoline and burning oil, and, in consequence, has a high fire test and is free from sediment and wax, it may be of a much higher value as a lubricant, requiring no refining. Such oils are sold as natural lubricating oils.

15. The refiner generally attempts to produce all, or a part, of the compounds given in Table II by fractional distillation and subsequent purification with sulphuric acid, caustic soda, and such other chemicals as the distillate may require.

The specific gravity being carefully taken, a given quantity of the crude oil is heated to 120° F. and poured into a graduated cylinder to settle. After standing for 10 or 12 hours, the amount and nature of the sediment is determined. Another sample of definite quantity of the crude is weighed and then dried for 24 hours over sticks of chloride of calcium. The difference in weight is ascribed to the loss of water, from which the percentage of water can be readily calculated.

The presence of certain offensive sulphur compounds may be detected by the odor, but they will be sure to appear in the distillates. The condition of these compounds is best determined by treating the distillate with a small quantity of sulphuric acid, decanting, washing the oil with water, neutralizing with caustic soda, and testing the sample with sodium plumbate, as described later.

16. Fractional Distillation Test.—For the fractional distillation test, a flask of about 150 cubic centimeters capacity, having an outlet tube about 7 centimeters above the bulb and extending downwards at an angle of 75°, as

shown in Fig. 1, is used. The observance of exactly these dimensions is essential in order to obtain uniform results. 100 cubic centimeters of previously dried crude oil is introduced and a thermometer fixed in the cork inserted in the mouth of the flask, so that the top of the bulb is on a level with the vapor-outlet tube; the vapor tube is connected with a Liebig condenser, and an Erlenmeyer flask is used as a receiver.



FIG. 1

17. The flask and contents are gradually heated with a Bunsen gas lamp; it is well to interpose a wire gauze between the flame and the flask in the beginning, but afterwards this should be removed and the naked flame employed. The heat is so regulated that from 2 to 2.5 cubic centimeters of distillate passes over per minute. The fractions usually taken are: up to 150° C., naphtha; 150° to 300° C., burning oils; over 300° C., residuum.

As soon as the thermometer indicates the temperature at which it is desired to cut off the fraction and begin the next, the temperature is permitted to drop 20° and then reheated to the original temperature. This is done as often as any appreciable quantity of oil is obtained before the fractionating temperature is reached. The first fraction having been completed, the second is taken in the same way. All over 300° C. is permitted to remain in the still, or it may be further divided into a secondary oil and a tar of 19° to 21° Baumé.

In the same manner, closer fractions may also be taken, as at each 50° C.; but in practice the manufacturer only

desires to know the fractions given above. The results will not agree entirely with those to be obtained on a larger scale, but are sufficiently accurate to give a comparative idea of the crude oil under consideration. The distillates should now be tested as to gravity and general purity.

18. Many practical refiners look upon such results as inaccurate. It must only be considered comparative and the refiners' business must determine what percentages may be obtained. In most cases, the refiner is able to increase his output of illuminating oil as much as 25 per cent. over the laboratory figures. If the operator in the laboratory, however, will observe the products distilling over above the 300° C. (572° F.), he will usually find that considerable oil in this fraction may be used with the illuminating fraction without injury to the color, though they are of much higher gravity.

19. The different States have different requirements, and if the sales department handles the products to the best advantage, it will be able to dispose of a large percentage of its heavy oils as illuminants of low fire test, or as high-fire-test railroad or lighthouse oils. The former may be accomplished by mixing with the heavy oil a fraction of the heavy naphtha distilling over just before the 150° C. cut. This produces a good burning oil, but low in fire test. If, however, the refiner wishes to sell "gilt-edged" illuminating oils of high gravity and fire test, he must dispose of his heavy oils in some other way. A low-fire-test illuminating oil is usually a better burning oil than one of high fire test, and without doubt many of our States have acted unwisely in placing the fire-test requirement so high (see State's requirements given later). Most foreign markets have a low-fire-test requirement, and this has permitted some refiners to produce as much as 80 per cent. of illuminating oil from Pennsylvania crude oil.

20. The tests given in Table V will be found accurate in the production of a good burning oil of 150° F. fire test.

21. In making a careful examination of the distillation tests given in Table V, it will be observed that the petroleum

TABLE V

Name of Oil	District From Which Sample Was Taken	Specific Gravity. Degrees Baumé	Boiling Point. Degrees Centigrade	First Fraction		Second Fraction		Third Fraction		Fourth Fraction		Sulphur	Water	Sediment
				Per Cent. by Volume up to 150° C.	Gravity of Distillate. Degrees Baumé	Per Cent. by Volume. 150° to 300° C.	Gravity of Distillate. Degrees Baumé	Per Cent. by Volume, from 300° C. Upwards	Gravity of Distillate. Degrees Baumé	Per Cent. by Volume of Residuum	Gravity of Residuum. Degrees Baumé			
Pennsylvania.	Emlenton, Pa., July 10, 1900	46.5	80	18.2	67.0	32.45	44.3	36.50	34.5	11.42	19.3			
Scio.....	Scio, Ohio, April 15, 1899	48.1	40	23.33	69.9	30.00	47.0	40.23	42.5	6.16	19.5			
Barnesville...	Barnesville, April 15, 1900	46.3	40	24.50	70.0	41.90	45.6	22.60	39.7	10.00	20.0	*		
North Lima...	Tiffin, Ohio, April 30, 1898	42.1	40	21.87	67.0	43.75	44.4	19.53	34.7	19.75	20.8	*		
North Lima...	Freeport, Ohio, April 30, 1898	41.0	70	17.60	60.0	41.60	44.3	17.70	39.0	22.30	19.7	*	1%	
North Lima...	E. Toledo, Ohio, May 14, 1898	35.0	80	10.71	59.0	31.25	44.3	29.35	38.5	22.67	19.3	*	3%	*
Indiana.....	Warren, Ind., June 10, 1898	33.0	83	4.00	65.0	28.50	45.0			67.00	25.3	*	5%	*
Texas.....	Corsicana, June 14, 1898	38.5	55	11.20	67.2	49.63	45.6	13.98	33.5	23.75	19.3			
Kansas.....		32.0	60	14.25	58.0	78.20				2.50	17.9			
California	Ventura County	28.0	60	9.70	60.0	29.10	40.0			61.20	17.0			
California	Fresno County	36.0		33.06	49.0	57.85	30.0			9.09	19.5	*		
Baku.....		32.0		8.50		40.70		18.30		20.00				
Baku.....	32% lubricating oil	28.0				26.00		10.00		10.00				
Baku.....		33.0		23.00		38.00				39.00				

* Indefinite quantities.

obtained from the different fields yields varying quantities of light and heavy oils. The first eight samples were carefully selected and all tested in precisely the same manner, so as to yield concordant results. The tests were all made for the purpose of giving certain manufacturers some idea of the value of these oils when distilled for commercial purposes. The other samples were taken by different authors; and doubtless there was some difference in the method of testing. The second and fifth samples have been distilled and refined on a commercial scale in the Sun Oil Company's refinery, when the same results were obtained.

22. In order to understand the value of such tests, let the Pennsylvania oil be taken as a basis and compare the other oils with it. In this way it will be seen how the practical refiner will be able to make a choice. The first three yield very nearly the same results and are, at present, rated under the head of Pennsylvania oil in the American crude-oil markets. The second and third were taken from new territory, and consequently yielded a large percentage of light oils. The second gave exceptionally fine "first" and "second-grade" oils of high gravity, and on that account received a premium in advance of the market price at the time these tests were made. Since the increasing demand for petroleum cylinder oils, the oils that are free from sulphur and have a light color and no sediment are manufactured into cylinder stocks. The Barnesville oil being of a darker color and not perfectly clear will not produce as fine a cylinder stock, and is, therefore, not valued as highly; but since the Pennsylvania crude stocks are decreasing, this oil enters the pipe lines and, being mixed with the other oils, passes unnoticed. The fourth oil given in Table V is a darker oil and contains an objectionable amount of sulphur.

23. In looking over the percentage columns of Table V, but little difference is found between the first North Lima oil and the preceding ones, excepting that it yields a larger percentage of residuum. The Tiffin field produced some of

the finest North Lima oil thus far obtained from the State of Ohio. As will be observed, its gravity is quite good; but since it contains sulphur in such combination which has been found difficult to remove, it is not of a high value. With the increasing demand for kerosene, paraffin, and other petroleum products, the processes for refining this oil have been constantly improved until the refiner is able to place it on the market in competition with products of the higher priced petroleum.

The prices of the Pennsylvania and Ohio oils have been gradually approaching each other and, doubtless, within a a very few years an oil similar to that from Tiffin will command the same price as that from the older fields in Pennsylvania. The fifth sample contains less light naphthas and more heavy oils.

The sixth and seventh samples are much poorer grades, being of little value to the refiner; the moisture and sediment are particularly objectionable. The principal use for oils of this kind is as fuel.

24. The Texas oil is comparatively free from sulphur, and is finding considerable use as crude oil to be refined. Its products are quite good, with the exception of the presence of asphalt.

The California oils have been examined of late by Professor Mabery. His reports show nitrogen compounds and considerable heavy oils. His vacuum distillations have brought out a fine line of lubricating oils almost entirely free from crystalline paraffin. Developments are now proceeding so rapidly in this field that these analyses are by no means sufficient to form a judgment of the crude oil of this State. The greater part of it is still finding its market as fuel.

25. In comparing the Baku petroleum with the other samples, it is necessary to take into consideration the fact that the oil is principally composed of the naphthenes, which are much less volatile than the compounds of the American petroleum. Redwood states that as much as 50 or 60 per cent. of the oil sold is residuum.

NATURAL GAS

26. Occurrence.—The history and development of the **natural gas industry** are so closely allied with that of petroleum as to be practically a part of it. As Doctor Orton has said, "They have a common history, produced from the same sources, accumulated by similar agencies, and stored in the same reservoirs. . . . Petroleum never exists free from gas, but it is sometimes asserted that gas is found that has no connection with petroleum. This claim is doubtful, and if the driest gas could be followed throughout its underground reservoirs, it is altogether probable that accumulations of oil would be found in every case."

27. The knowledge and use of natural gas in America antedates that of oil. **Burning springs,** as they were called, were observed by the earliest settlers. Nearly all of the salt wells, besides containing oil, produced some gas; the gas from the salt wells along the Muskingum was utilized in evaporating the brine. It was encountered in drilling the early oil wells, and was looked upon as a nuisance.

28. Uses of Natural Gas.—Fredonia, New York, has the distinction of being the first town to use natural gas as an illuminant; in 1824, it was used to illuminate a mill and a number of houses in the town. During the early operations on Oil Creek, in 1862, it was used as fuel in firing the boilers in pumping wells.

In 1867, it was piped to some houses in Oil City, where we have the first record of its being used for cooking and heating dwellings. The first attempts were not considered very successful on account of the changing pressure, but this objection having been overcome, it was regularly installed for such purposes. It has since held the distinction of being the most convenient and economical fuel to be had for domestic use. The cities and towns about the oil fields were quick in adopting its use. It was also found to be valuable in the furnaces of iron mills and glass factories.

29. When the "oil fever" had taken possession of the people throughout the country, on account of the successes on Oil Creek, the earth was punctured in almost every quarter of the United States, and it was soon observed that gas was more widely distributed than oil.

30. The celebrated Harvey well, in the southern part of Butler County, Pennsylvania, was struck in November, 1874, at a depth of 1,145 feet. It was located between abrupt hills in a valley about 300 feet wide. The gas was conveyed a distance of 150 feet in a 6-inch iron pipe, from which it discharged with the force of steam. The gas was allowed to burn for some time, as it escaped in a flame 40 feet high and 15 feet wide.

In the Fall of 1875, Spang and Chalfant piped it to their rolling mill at Sharpsburg, a distance of 17 miles. The Burns well on the Duffy farm, near St. Joe, in the same county, was struck in 1875 at a depth of 1,600 feet. The output of this well averaged 12,000,000 cubic feet of gas per day. It was piped to Freeport, where it was used for fuel and illuminating purposes. For heating purposes it was considered 25 per cent. better than bituminous coal.

The Delamater well in the same county was an oil producer so long as the owners did not go below the third sand. When they did, they lost a 10-barrel well and struck what appeared to be an inexhaustible reservoir of gas, although the volume of gas was not so large as that of the Burns well, one-half mile distant. This well was also connected with the line leading to the Sharpsburg rolling mills.

Although the manufacturers of Pittsburg knew of the value of the gas in the oil regions, they did not make any use of it for 20 years or more. But finding that a better quality of iron could be produced at about two-thirds the cost, it was then readily adopted. Glass factories, also, began to use it, and found that a better quality of plate glass could be made.

In 1877, the Haymaker well opened the Murraysville field, and was piped to Pittsburg by Pew and Emerson. The Philadelphia Gas Company procured large tracts of land and sent their product to Pittsburg.

At East Liverpool, Ohio, the gas was used as a substitute for coal in the early seventies. It has been one of the causes for the building up of one of the most prosperous pottery industries of America.

31. The discovery of high-pressure gas in the Trenton limestone belongs largely to Dr. Charles Oesterling, of Findlay, Ohio. Natural gas had been known in Findlay since the country was first settled. In digging wells, cisterns, and sewers, in springs and rock crevices inflammable gas had been found. It had been utilized in a small way for a number of years. Gas was first used there as an illuminant by Daniel Foster, who introduced it into his house in 1838. But Dr. Oesterling seems to have been the first one to see that this source of light and heat could be utilized in a large way. For a number of years he urged the formation of a stock company to drill for gas. But it was only after Pittsburg had demonstrated the value of this fuel to manufacturing industries that it was possible to carry out such a project. The first well was put down in November, 1884; the gas from it came with sufficient force to run an engine when turned into it. The Karg well was struck in January, 1886. It is claimed to have been the largest "gasser" in the Findlay district. Its estimated output was 12,000,000 cubic feet per day. The Trenton limestone gas resembles the oil from this source in containing odorous sulphur compounds.

32. Composition of Natural Gas.—The following is the analysis of Findlay gas as made by Prof. C. C. Howard :

Marsh gas	92.61%
Olefiant gas	.30%
Hydrogen	2.18%
Nitrogen	3.61%
Oxygen	.34%
Carbon dioxide	.26%
Carbon monoxide	.50%
Hydrogen sulphide	.20%

33. The large amount of gas that was found in Ohio led to the drilling of wells in Indiana. The gas found here, however, has been principally utilized for domestic purposes and the manufacture of plate glass. In Kansas, where new gas fields have recently been opened up, the gas is being utilized in smelting the zinc ores from Missouri. Among the other States in which gas has been found in large quantities might be mentioned Kentucky, Tennessee, Texas, and Michigan.

34. The waste of this valuable fuel within the borders of the United States can hardly be estimated. Jets of gas 10 feet across and 30 feet high were permitted to burn undisturbed for years, houses were heated to such an extent as to make it necessary to open doors and windows in mid-winter in order to make them comfortable, and street lamps are still burning day and night in some towns.

THEORIES OF ORIGIN OF NATURAL GAS

35. Organic Theory.—The theory that petroleum is of organic origin is strongly supported by many American and European scientists, but the adherers of this theory do not agree as to the point whether petroleum is of animal or vegetable origin. Lesquereux and G. P. Wall are of the opinion that petroleum is due to the decomposition of marine plants, just as coal is the result of the decomposition of terrestrial vegetation. In contradiction to this, others claim that it is of animal origin and support their theories in many ways. Dr. Orton has made a very careful study of the subject, and his ideas, which are probably correct, may be summed up as follows:

1. Petroleum is derived from organic matter.

2. Petroleum of the Pennsylvania type is derived from the organic matter of bituminous shales and is probably of vegetable origin.

3. Petroleum of the Canadian type is derived from limestone and is probably of animal origin.

4. Petroleum has been produced at normal rock temperatures (in the American fields at least) and cannot be considered a product of the destructive distillation of bituminous shale.

36. Inorganic Theory.—Among the supporters of the inorganic theory are such eminent scientists as Berthelot, Mendeleeff, and Moisson. The latter materially extended the field of information in regard to carbon and its compounds and its reaction with metals, and advanced a theory based on the reaction of water with certain metal carbides. He says the reaction that appears to us most wonderful is the ease by which one can produce gaseous, liquid, or solid hydrocarbons by the action of cold water on certain metal carbides. The action of water on aluminum carbide gave pure methane and might be the cause of natural gas issuing from the earth in certain localities. A similar reaction may serve to explain the formation of liquid hydrocarbons or petroleum; in one case, 100 grams of liquid hydrocarbons was received from 4 kilograms of uranium carbide. To a great extent this mixture is composed of the ethane series, and only a very small percentage belongs to the acetylene series. At the same time, under ordinary pressure considable hydrogen and methane were obtained. This led to the belief that under pressure we would have nothing but methane or saturated series similar to petroleum. Doubtless during the early period of the world's existence the entire carbon was combined in the form of metal carbides. A sudden movement of the earth's crust would permit water to come in contact with these metal carbides and produce a sudden evolution of gas. The temperature would rise and produce polymerization of the hydrocarbons, and thus produce a very complicated mixture of these compounds. Through existence of metallic carbides, produced so readily by means of high temperatures, which are doubtless found in the earth's interior, one could readily account for the existence of gaseous, liquid, and solid hydrocarbons.

37. In giving a short review of these various theories and opinions, it is not the purpose to support one or the other, but simply to present them to the student as they now stand. A careful study of the subject, however, leads to the conclusion that the formations are, in all probability, not of a single origin, but that, for most instances, the views advanced by Dr. Orton are doubtless correct.

THE PRODUCTION OF CRUDE OIL

OIL WELLS

38. Location.—The drilling of wells is now systematically done by the most approved methods. The right to operate having been obtained (usually for a certain percentage of the gas or oil obtained), the well is sunk according to the opinion of the operator, although due consideration is paid to the wishes of the landowner.

In the early operations along Oil Creek all locations were made along the streams and ravines; as the hills were considered too high to produce oil, it was not until about 1870 that wells were drilled on the hillside as well as in the valleys. This soon developed certain defined lines known as *oil belts*, which have governed nearly all locations in Pennsylvania and West Virginia.

39. Construction of Rig.—The location being selected, the next thing in order is to put up a *rig*, or *derrick*. The first derricks were made about 34 feet high and were completely covered. The modern rig, as will be seen in Fig. 2, is of very simple construction, but possesses the necessary strength. Four strong uprights (2″ × 8″ beams spiked together) are held in position by cross-braces, and rest on heavy wooden sills. It is usually 82 feet high and 12 feet square at the base, the height depending on the depth to

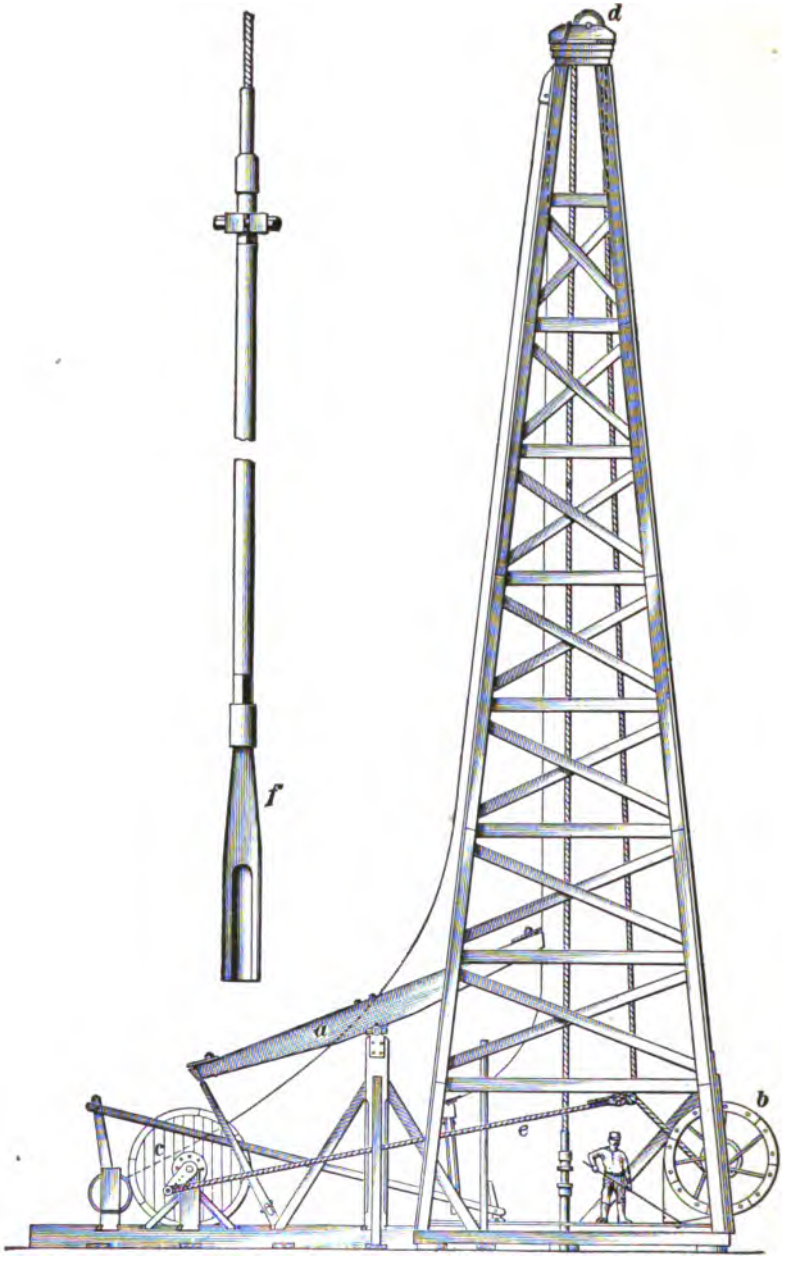


FIG. 2

which it will be necessary to drill. In shallow-well territory, a shorter rig will materially reduce the cost of operation. Connected with the rig is a long, shed-like structure, known as the *belt house* and *engine house*. This contains the engine and band wheel *c*, which communicates its motion through a pitman with the walking beam *a*.

It is customary for the owner of the well to furnish the necessary steam power; that is, a 25-horsepower boiler and a 12- to 15-horsepower engine, while the contractor furnishes the drilling tools, cable, blacksmith's outfit, and fuel for the boiler. Contracts are made at a certain price per foot, which varies according to the nature of the territory.

The old hollow sycamore that was used by the Ruffner Brothers in the first rock-bored well gave way to a smaller conductor, made of 2-inch planks nailed together to form a square 6 or 8 inches on the inside. A hole was usually dug to the bed rock, and this conductor placed in position as the rig was being built. This, in turn, has given way almost entirely to the drive pipe, which was first used by Drake in his well on Oil Creek. The first thing for the driller to do, after the engine has been set and all necessary connections have been made, is to drive an 8-inch or 10-inch iron pipe to the bed rock.

40. Drilling Tools.—This having been accomplished, the well is started by what is called the **spudding process.** It is usually carried out with light, short tools and a large bit, shown at *f*, Fig. 2, about the size of the internal diameter of the drive pipe. The tools are fastened to a short cable, which is passed over the crown pulley *d* and fastened to the *bull wheel b*. Now, by fastening a *jerk rope e*, which is attached to the crank of the band wheel, to one end of the cable, the tools may be raised and dropped at each revolution of the wheel. When sufficient depth has been reached by this operation to admit the use of a full string of tools, the spudding machinery is abandoned.

A good cable $2\frac{1}{8}$ inches thick and 3,000 feet long (in deep-well territory) is passed over the crown pulley and coiled on

the bull wheel. The .end of the cable is secured in the rope socket, which is then attached to the full set of drilling tools, and these swung into the derrick. Their entire length occupies the space from the crown pulley to the derrick floor, and their weight is over a ton. They consist of a rope socket, 3 feet 6 inches long, weighing 80 pounds; 5½-inch jars, 7 feet 4 inches long, weighing 320 pounds; augur stem, 50 feet long, weighing 1,800 pounds; bit, 5 feet long, weighing 300 pounds.

The augur stem varies according to the nature of the country in which the well is being drilled. Formerly all drilling was done with a center drill, and the hole enlarged and irregularities smoothed up by means of a rimmer. Now, all drilling is done with a fluted drill, having a rather blunt point. By this change, the time of drilling has been reduced over one-half.

41. Everything having been carefully examined and adjusted, the regular drilling commences, and the machinery is kept in motion day and night. One driller and his helper, the tool dresser, work from noon until midnight, and another pair from midnight until noon.

The driller watches the jar, from which he can tell what his drill is doing. By means of the temper screw he lowers the tools and penetrates the rock; when the whole length of the screw has been run out, or the slow progress of the drill gives warning that it is working in hard rock and needs sharpening, he informs the tool dresser that he is ready to "draw out." The tools are withdrawn, a new bit put in place of the dull one, and the sand pump run down to remove the water and sediment that has been caused by the loosening of the earth and rock by the drill. This being accomplished, the bit is tightened with the wrenches and the tools lowered to continue as before.

While the driller is attending to his work, the tool dresser looks after the boiler, places the dull bit in the fire, and attaches a rope to the bellows handle; this rope is attached to the wristpin of the band wheel so that the fire is blown

and the bit heated without further attention. Having oiled his machinery and attended to other odd duties, he returns to his fire and closely observes the "heat." As soon as the proper heat has been obtained he calls the driller, hands him a sledge, and together they hammer the end into the proper shape. The whole operation requires a thorough knowledge of blacksmithing, for the bit must not be overheated while in the fire and must be properly tempered after it is dressed. The entire work is such as to require strong, healthy, and experienced men.

42. Casing the Hole.—In order to prevent the water entering the oil strata, the well is cased as soon as the driller has reached the bottom of the water-bearing rocks. In some instances it is found necessary to put in several strings of casing on account of different veins of fresh and salt water. As soon as the casing is placed the drilling continues, but with a smaller sized bit—one that will enter the casing.

Before wells were cased, it was frequently almost impossible to tell whether or not an oil-bearing stratum was reached when drilling was suspended. If it was considered worth testing, a seed bag was put on to the end of the tubing and lowered into the well and permitted to stand several days. The water was then exhausted from the bag and the oil permitted to leave the rock. This method, however, was objectionable, for the seed bag was apt to burst and undo the whole work at the first sign of oil. Mud veins in the third sand on Oil Creek and at Pithole would stick the tools effectually before the wells were completed; but casing has proved an entire absence of anything like mud veins near the oil strata.

43. While the general principles concerned in the operation of drilling and in the construction of the tools have not been materially modified, labor-saving devices have been steadily introduced. Wells are now drilled in 15, 20, or 30 days, that in the beginning of the oil industry would have required as many months to complete. Great ingenuity is displayed by a professional well driller in contending with and successfully overcoming the innumerable obstacles,

mishaps, and breakdowns with which he meets. The tools that have been invented on the spur of the occasion to meet some unexpected difficulty are to be numbered by the hundred. They are of all kinds—from the delicate grab designed to pick up a small piece of valve leather, or a broken sucker-rod rivet from the pump chamber, to the ponderous string of pole tools containing tons of iron, which, at a depth of 1,500 feet or more, can unscrew a set of stuck tools and bring them up piece by piece, or cut a thread upon the broken end of a sinker bar or an auger stem, so that it can be screwed fast to and loosened by the use of "whiskey jacks" at the surface.

44. Location of Strata.—In the early days of oil operations the dip of the rock was confusing to the driller. He could not understand why, if oil was found at a depth of 69 feet at Titusville, Pennsylvania, it could not be found at the same depth at Bradford, Oil City, and Butler. In fact, the territory around Bradford was condemned because oil could not be found at shallow depths. The Barnsdall well, which had been drilled to a depth of 200 feet in 1862, was deepened to 875 feet in 1866 without success. The driller once realizing that there were a number of sands and they were not to be found at the same depth throughout the country, proceeded to study this matter, and today is well informed regarding his particular work. His key rock in Western Pennsylvania is the limestone, which is from 5 to 25 feet thick in Butler, Armstrong, and Clarion Counties.

The following is an extract from a letter by a practical driller regarding his manner of estimation in the Butler County oil field:

"The limestone, which varies somewhat with the surface and location of the well, is used as our guide. The Butler County gas sand, commonly called **Berea grit,** is 875 feet below the surface of the limestone. This is the first oil-bearing sand of value in this section, with the exception of a few odd wells producing small quantities of pale oil from the 60-foot, which is from 50 to 65 feet from the limestone.

This is the sand that was formerly worked for brine. Following the Berea grit is what is known as the '100-foot,' which is about 1,000 feet below the surface." Table VI gives the different formations as they are known to the practical driller.

TABLE VI

Name of Sand	Distance Below Surface of Limestone. Feet
30-foot	1,150
Blue Monday, or Snee sand	1,200
Boulder	1,230
Third sand	1,250
Fourth sand	1,350
Fifth sand	1,425
Speechly	2,325

These depths, however, vary somewhat; in Washington County, for instance, the fourth sand was only reached at a depth of 2,640 feet, while in Armstrong County, the fifth sand was reached at about 2,000 feet, and the Speechly sand at about 3,000 feet. The latter is the productive oil sand of the Bradford field, where it was found at a depth of about 800 feet.

45. A drilling record of a well located in the Battelle District, Monongalia County, West Virginia, is shown in Art. **47.**

46. The Gordon sand of Washington County, Pennsylvania, and the West Virginia field is the fourth sand of the Butler-Venango field. This territory of deep and expensive wells has yielded some wonderful returns. But the expense has always retarded the developments. Only companies with extensive resources are able to hunt for new pools. A well in northern West Virginia costs about $9,500; in Washington County, Pennsylvania, about $8,000; in Butler County, $3,000; while on Oil Creek, many wells were put down for less than $500.

47. Torpedoing.—This method of adding to the productiveness of oil wells by the use of explosives is wholly American. The process was patented by Col. E. A. L. Roberts, who believed that the oil was contained in crevices

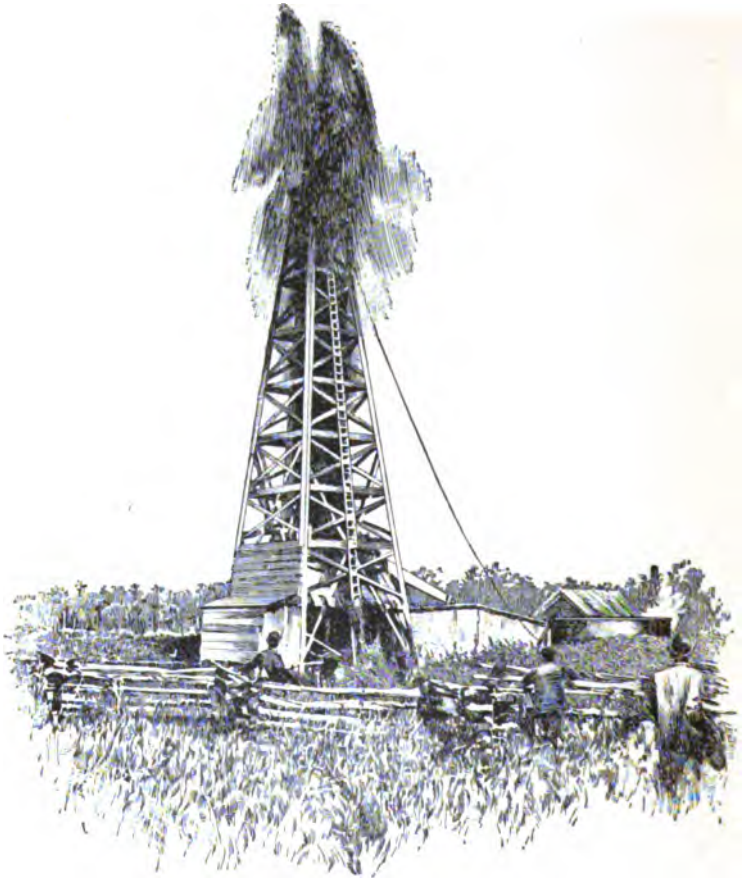


FIG. 3

not always reached by the bore hole. He proposed using nitroglycerine, or some other explosive, in the well to break up the rock and open these pockets. His idea met with disfavor at first, but he was finally permitted to make an

attempt upon what was known as the "ladies' well," at Titusville, Pennsylvania, and obtained favorable results. He next tried it on the "Woodin" well, a dry hole on the Blood farm. His first torpedo secured a production of twenty barrels per day; within a month, a second torpedo was tried, which brought up the production to eighty barrels.

- The most wonderful results ever produced by a torpedo, however, was that of the Armstrong well on Thorn Creek, Butler County. Fig. 3 represents an oil well shortly after shooting.

Veins Met	Distance of Top of Vein Below the Surface. Feet	Distance of Bottom of Vein Below the Surface. Feet
Pittsburg coal.....	1,306	
Dunkard sand.....	1,815	1,860
Gas sand.....	1,970	2,110
Salt sand.....	2,200	2,240
Indian sand.....	2,590	2,630
Squaw sand.....	2,800	2,920
Thirty-foot sand.....	3,080	3,100
Gantz sand.....	3,170	3,175
Fifty-foot sand.....	3,205	3,305
Gordon sand.....	3,512	3,538

THE TRANSPORTATION OF OIL

TRANSPORTATION OF CRUDE OIL

48. Oil Barrels. — The early method of transporting crude oil in America was in 42-gallon barrels, which were filled at the wells and hauled to the nearest railway station or wharf along the river. Oil Creek was used to such an extent that the amount of water in it was not adequate to carry the boats containing the oil. The shippers, however,

resorted to a system known as *pond freshets*. A dam at the saw mill near Drake's well stored the water, which at the appointed time was permitted to escape, and carried the loaded boats to Oil City. Skilled pilots assumed the responsibility of delivering the oil to the larger boats at Oil City for conveyance to Pittsburg, at from $100 to $200 per trip. The teamsters and wagons were in such demand that a man with two stout horses could earn $20 per day. With continual use, the roads became a canal of mud from 3 to 4 feet deep. The hauling of a few hundred barrels not infrequently consumed so much time that the shipper, through the rapid fluctuations of the market, would sometimes not realize enough to pay the wagon freight.

The following is given by McLaurin as the expense per barrel of oil delivered at New York in January, 1866:

Government tax	$1.00
Barrel	3.25
Teaming from Pithole to Titusville	1.25
Freight from Titusville to New York	3.65
Coopering and platform expenses	1.00
Leakage	.25
	$10.40

49. Tank Cars.—Flowing wells soon taught the producer that the barrel system was not adequate to handle the crude in such cases. The Empire well alone produced more oil in one day than all the coopers of the nearby cities could produce barrels for in a week. The railroads laid switches and branch lines to the places that contained rich pools, and for transportation placed large wooden tanks, holding about 2,000 gallons, on flat cars. In 1871, these wooden tanks were replaced by cylindrical tanks made of boiler iron, similar to those in use today. Yet this still necessitated the hauling of oil several miles before the railroad was reached.

50. Pipe Lines.—In order to overcome this difficulty, pipe lines were proposed. The first successful one was laid

between Pithole, Pennsylvania, and the Miller farm. This system was held in such disfavor, however, that it was only by a rigorous enforcement of law that the interference of teamsters, etc. was overcome. Other pipe lines were shortly laid to this point and loading racks *a*, Fig. 4, constructed, which enabled several men to load a train of tank cars in a few hours. The same system is in vogue today for short shipments and the transportation of refined oil.

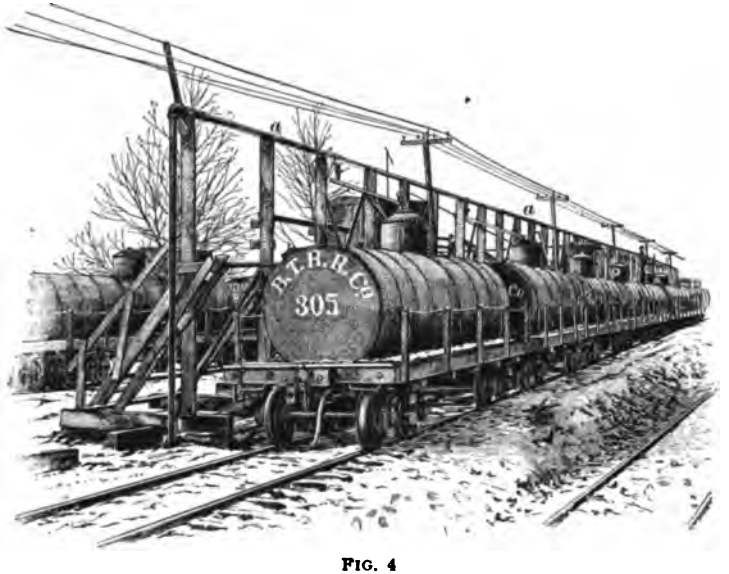


FIG. 4

Consolidation soon followed, and trunk lines were established. In 1875, a 4-inch pipe line about 60 miles in length was laid from the lower field (Butler County) to Pittsburg. The Bradford field needed especial attention on account of the large output and heavier crude. Improvements in pipes were made, and the first 6-inch line was laid from Tarrport to Carrollton in 1879. About the same time the first long-distance line was laid to Cleveland, and a trunk line to the eastern seaboard begun. At present, the oil field is a network of small lines, while several trunk lines traverse the whole length of New York and Pennsylvania, delivering

thousands of barrels of oil daily to refineries, and loading tracks en route and at their termini at the seaboard. Refined oil, as well as crude oil, is now made in Cleveland, Oil City, and a number of inland towns, and transported through pipe lines to the seaboard, where it is loaded into immense tank steamers and sold in foreign markets.

51. The pipe-line system has been the means of saving millions of barrels of oil. When the drill approaches the sand, the pipe line is connected to the tank to take care of the production. In the case of some of the largest wells ever struck, the well owners were only provided with a 250-barrel tank, and it was only because the pipe-line company placed a large pump in position and forced the oil into the large mains, that the production was saved. Improvements in this system have been no less wonderful than in many of the other branches of the industry. The finished products are transported in tank cars to the large stations, from which they are usually sent out in barrels to the retailer.

TRANSPORTATION OF REFINED OIL

52. In order to accommodate the small jobber as well as large retail dealers, the refiners and railroads have compartment tank cars, as shown in Fig. 5 (this car has three compartments, each having a dome, as shown at *A*, *B*, and *C*). In such a car may be shipped kerosene, gasoline, and benzine, or different grades of kerosene. When oil is shipped in tank cars, the shipper is certain that the goods will arrive in the same condition as when shipped from the refinery. For short shipments of smaller quantities, everything is shipped in barrels. These barrels are usually of 50 gallons capacity and are made of the best oak staves.

53. Preparation of Barrels.—In order to render the barrels capable of holding the oil without leakage, they are coated internally with glue, about 1 pound of glue to four barrels being required. The hot-glue solution is poured into the barrels, the barrel bunged up with a temporary bung,

and rotated so as to coat the entire inner surface; the surplus is afterwards drained out.

The glue having become hard, the barrel is painted with a cheap paint composed of a mixture of the cheapest color, benzine, and rosin. The rosin gives the barrel a hard, glazed surface and good appearance. Barrel painting is a process that requires considerable skill to do good work with the least loss of paint. A first-class painter is capable of

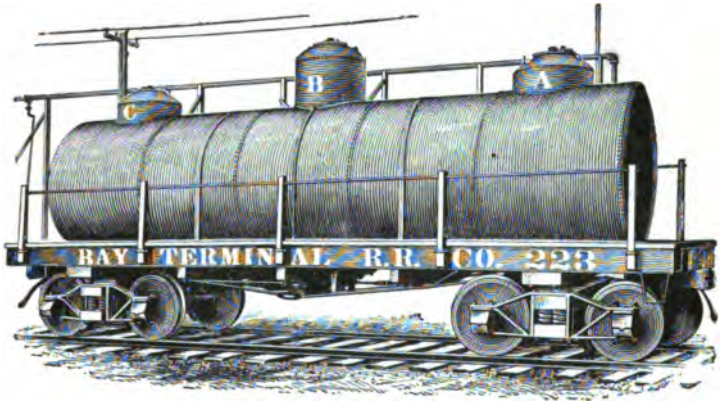


FIG. 5

painting a barrel per minute. In order to avoid the taint often caused by the glue of the barrel or leakage of the light gasolines, iron barrels are sometimes used. These, however, are only used for short shipments, where they can be returned promptly, since the barrel is too expensive for the receiver to own or for the refiners to lose.

At the present time all cities and larger towns are supplied in bulk and distributed by means of tank wagons, thus avoiding loss or contamination by barreling.

54. Shipment in Tin Cans.—Shipments of oil to warm climates or to distant parts are made exclusively in cans packed in cases. This avoids leakage and cheapens transportation. The cans are rectangular in shape, hold 5 gallons each, and are put up by twos in rectangular wooden cases.

PETROLEUM AND PRODUCTS

(PART 2)

REFINING PETROLEUM PRODUCTS

THE REFINING OF ILLUMINATING OILS

1. Introductory.—In spite of the fact that Samuel M. Kier produced a very poor illuminant when compared with the kerosene of today, yet he has the distinction of being the first to distil petroleum to be used for illuminating purposes. His "carbon oil," as it was called, sold for $1.50 per gallon; and by this means he disposed of all the petroleum the salt wells at Tarentum could produce. His operations, however, were of such a nature as to have but little influence on the industry when the crude oil appeared in abundance. The distillation of coal in retorts, and the manufacture of illuminating oils therefrom, which preceded the oil industry by but a very few years, was the real precursor in the development of the practical disposition of these enormous supplies of hydrocarbon oils.

2. Stills for Refining Petroleum.—In the refineries of the earlier days the stills were small, varying in capacity anywhere from 25 to 75 barrels. Many of these small stills are still in use, but stills constructed at the present time are much larger. The stills to be found in a refinery today belong to either of two models, viz., the *cheese-box* or the *boiler still*. The cheese-box still has a cylindrical body, flat

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steel-plate bottom, and dome-shaped top with outlet pipe at its highest point. They vary from 20 to 30 feet in diameter, and from 8 to 9 feet high. The use of this form of still is gradually diminishing on account of the longer time required in distillation and liability of accidents in the opening of seams in its bottom.

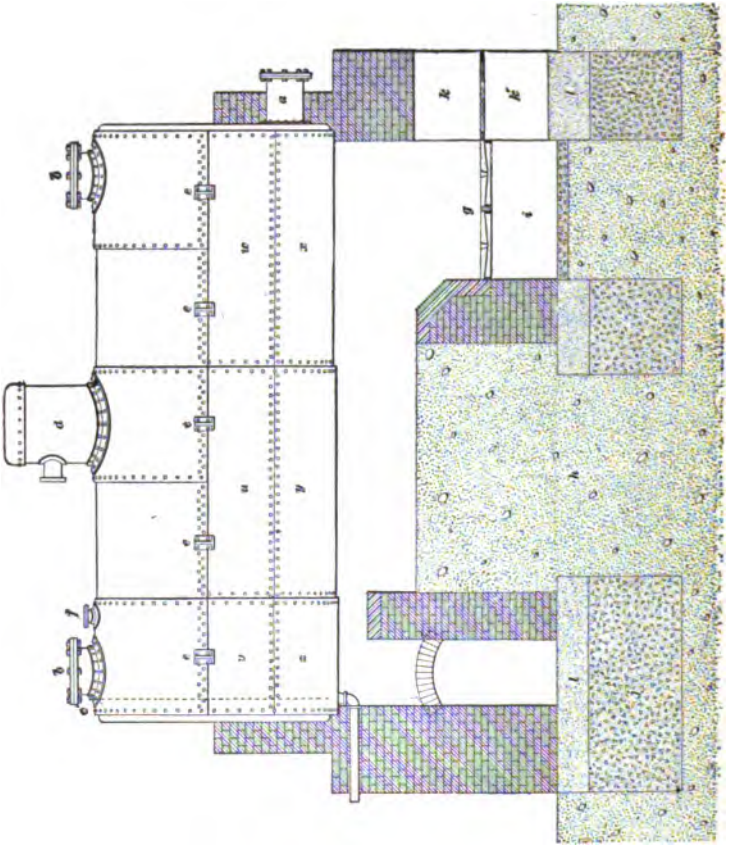


FIG. 1

3. Boiler Stills.—By far the most common still in use today is the horizontal cylindrical, or boiler type, shown in Fig. 1. The specifications for a 600-barrel boiler still are as follows:

Size of Stills: 12 feet in diameter and 30 feet long.

Dome: *d* to be 3½ feet high by 3½ feet in diameter and to have flat head.

Size of Material: Dome heads to be built of ½-inch flange steel, flanged. Dome shell to be built of ⅜-inch flange steel. Shell of still to be built of ⅜-inch flange steel. Bottom and side sheets to be built of ¼-inch firebox or still-bottom steel. Heads of stills to be flat and built of ½-inch flange steel, flanged. Bottom of still to be constructed of three sheets; two, *x* and *y*, each 12¼ feet long and 8½ feet wide, and one, *z* (the rear sheet), 6½ feet long and 8½ feet wide. Side sheets *w*, *u*, and *v* to be 6½ feet wide and 6½ feet long. Shell to be made of five sheets, equal lengths, and of such width as to connect with the side sheets on each side of the still.

Rivets and Riveting: All rivets to be ¾-inch iron, Burden make. All bottom and side sheets and dome to be double staggered-riveted. Shell and heads to be single riveted.

Connections and Man Heads: The still is to be provided with one 18-inch wrought-iron neck man head *a* on the front end, having faced ring and plate and square-headed bolts and cover crane, the man head to be located in the center of head and about 15 inches above the bottom of the still; two 20-inch cast-iron man heads *b* with hinged bolts, faced plate, and top, each to be riveted on center line of top near each end; one 15-inch wrought-iron flange riveted to rear of side of dome, about 3 inches below the top of same, for condenser connections; one 6-inch wrought-iron flange *f*, for filling connections riveted on center line of top of still, about 6 inches forward from rear man head; one tar valve complete on bottom of still near the rear end, and having stem *c'* extending up through center line of top of still about 6 inches forwards from rear end of the same, having wrought-iron flange and stuffingbox.

Stayrods, Lugs, Etc: Each still is to be provided with 10 cast-iron brackets *e*, 5 on each side, same to be riveted on

side of still, for the carrying of the still on the walls. Still to be provided with the necessary T bars and cross rods between heads to hold the heads firmly in place. This is to be accomplished by one T bar across the center of each head and by two 1¼-inch rods, with turnbuckles, running the full length of still. Also short T rods to be located above and below center T rods, having tie-rods extending from these and connecting with the sides of the still.

4. Stills are placed on good cement foundations *l*, Fig 1, and substantial brick walls; *g* are the grate bars; *k* is the furnace door, and *k'* is the door to the ash-pit *i*. The parts *h* are filled in with earth. The concrete *j* supports the cement foundation. About one-half of the still is exposed to the air (except for a sheet-iron jacket), to prevent burning of the vapor when it comes in contact with the walls of the still. The part exposed to the direct heat of the firebox should not exceed one-third the circumference of the still (12 feet, 6 inches). This is important, in order to protect the bottom of the still, since the level of the oil will come below that point at the end of the distillation and the iron will become overheated and warp. This will loosen the rivets and shorten the life of the still. Another important point is to guard against burning the vapor when the fire strikes above the oil line. In a well-constructed still all these points have been carefully considered. To the flange on the dome is attached a 10- or 15-inch vapor line. This vapor line leads to the condenser, at an angle of 65° to the surface of the still. Before entering the condenser it is customary to provide the line with a pressure and vacuum safety valve.

5. Condensers.—Condensers of many different types are in use; the most common form, shown in Fig. 2, is that in which the vapors traverse parallel pipes *a* of large condensing surface. The large 10-inch vapor line is divided into 4 4-inch pipes as it enters the condenser. The worm condenser is usually employed, but the old spiral form has given way to a worm composed of straight tubes connected

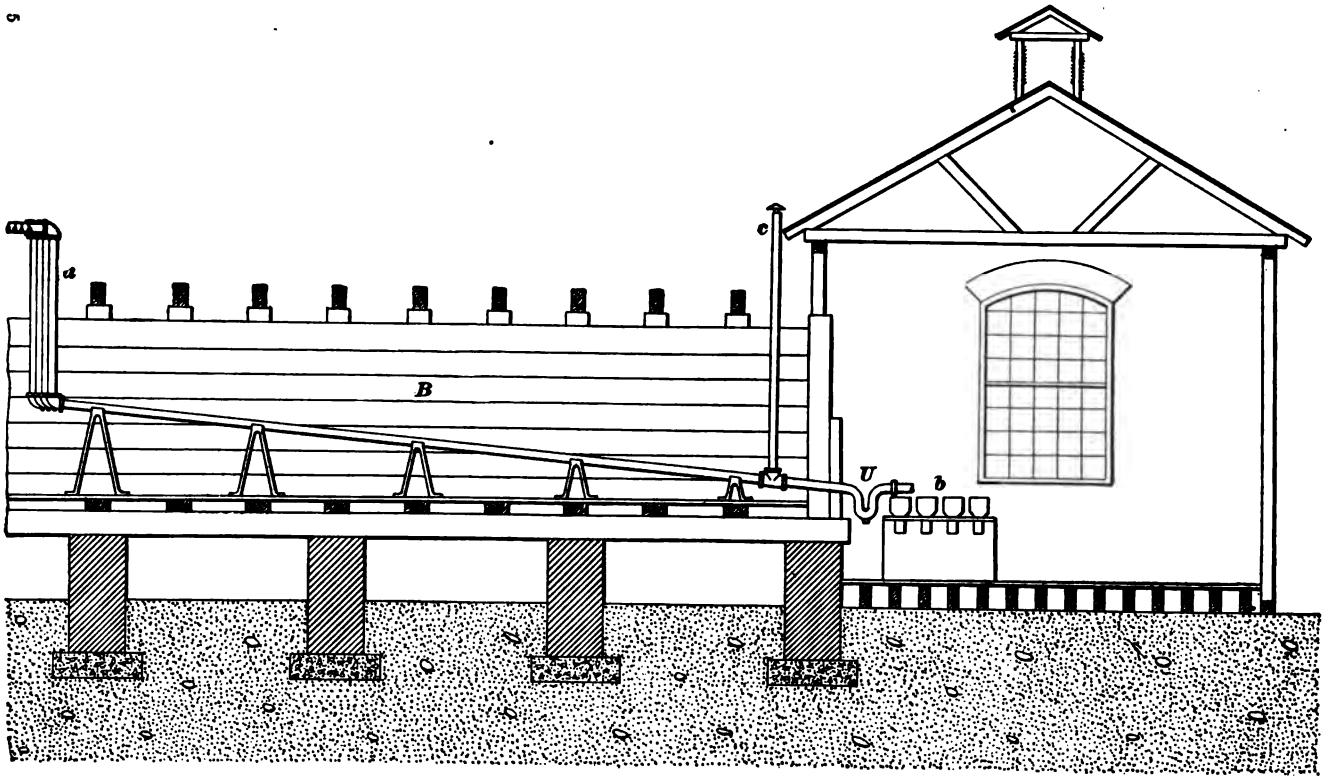


FIG. 2

by means of returns or elbows. These tubes or pipes are arranged in four parallel coils, with sufficient fall to prevent any condensed fluid from choking the passage within a rectangular tank *B*, made of iron or wood.

The common condensing medium is cold water. The water tanks are frequently large enough to accommodate a number of stills; the most convenient arrangement, however, where sufficient cold water can be obtained, is to provide each still with a separate condenser. The condensing surface required varies, of course, with the size of the stills, the grade of crude oil, and the products desired. For a 600-barrel still to be run for illuminating oil, where a large stream of cold water is at command, a condenser box, 40 feet long, 8 feet wide, and 6 feet high, containing a coil of 720 feet of 4-inch pipe and 576 feet of 3-inch pipe, has been found to be of the proper capacity. The supply of water should enter at the lowest point and the heated water leave the box by an overflow pipe.

6. The percentage of loss in the distillation depends in a great measure on the efficiency of the condenser. The water supply should be such as to admit the use of large quantities when necessary. It should be so regulated that even during the hottest summer weather all light distillates and illuminating oils do not pass the receiving house at a temperature exceeding 80° F. Formerly it was thought best to permit the water in the condenser to become heated while running off the illuminating oils. It was claimed that by this means distillates of a high fire test were obtained. The disadvantage of such an arrangement will be readily seen when we consider that the advantages are gained at the expense of a large loss of non-condensed light oils. At this period of sharp competition, refiners find that condensation must be as nearly perfect as it is possible to obtain in this way. Before the liquid leaves the condensing-pipe system to enter the receiving box, it passes into a **U** tube (see *U*, Fig. 2). The **U** tube forms a seal that prevents the passage of uncondensed gases.

In order to keep these gases from entering the tailing or receiving house, the pipe conveying the distillate has an upright pipe *c* attached before it reaches the building. The U tube prevents their passage, and the upright pipe discharges them from the line. These gases may be used as fuel, and in that case are drawn to the furnace by means of a steam jet at the point where they are to be used. This is of considerable economic value when operated properly, since fresh crude oil contains large percentages of gas dissolved in the oil, and these are always driven off with the first distillation; and again in the final distillation and coking process the heavy hydrocarbons are broken up and gases formed that cannot be condensed by ordinary means. These gases are an entire loss unless they can be used as fuel at the plant.

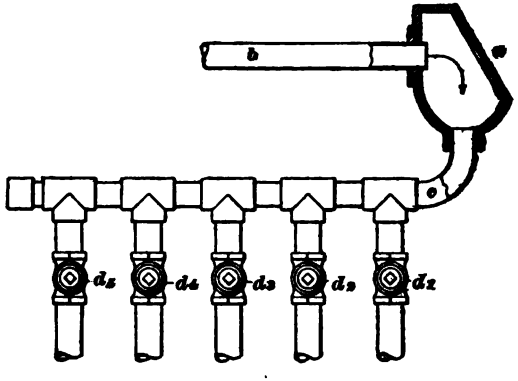


FIG. 3

7. After passing the U the oil enters the separating box *b*, which is usually provided with an automatic water tap that separates the water formed by the condensed steam and permits the oil to pass to the distillate tank. Fig. 3 shows the common separating box used in most refineries in the United States. It is a triangular-shaped, cast-iron box with a glass door *a*. The oil enters through *b* and passes out through *c*. Through the glass door the stillman observes the color and the size of the stream, and regulates his fires

accordingly. From here, samples are taken and the gravity of the distillate calculated. The stop-cocks d_1–d_6 communicate with the receiving tanks that are usually under ground. The oil having reached the gravity at which it is to run to some other grade, the one stop-cock is closed and the other opened.

8. Receiving Tanks.—The Canadian method of constructing these underground tanks is to nail together in a cylindrical form pieces of 1-inch lumber about 18 inches long and 4 inches wide. These pieces are fitted together to almost fill the hole that has been dug. The earth being a blue clay is tamped solidly about this structure, and after being water-soaked from without and oil-soaked from within, makes a perfectly impervious tank. The tank is not affected by the temperatures of the seasons, and requires very little attention or repair, the only part affected by age being its roof.

9. Storage Tanks.—Most refineries are provided with large storage tanks for crude oil, in which it is permitted to settle in order to free it from water and other sediment. These tanks are sometimes located on an elevation, and permit the still to be charged by gravitation. More commonly, however, they are situated on a level with the works, or even under ground, and require large pumps to force the oil into the stills. To allow for expansion of the oil on being heated, the still is usually filled only three-fourths to four-fifths full of crude oil. The cylindrical still previously described is charged with 525 barrels of Pennsylvania crude oil.

10. Distillation of Crude Oil for Naphtha, Illuminating Oils, Etc.—The man heads and all lines connected with the still are carefully examined and closed. The fire in the furnace being kindled, is at first kept low in order to permit the fixed gases and moisture to pass off slowly. If moisture is present, it is recognized by a bumping in the

still, becoming very severe if the firing is forced. With gentle firing, the oil is agitated without causing any injury, and the lightest gases and the water readily pass over through the worm. The quantity of water constantly increases until the water in the still boils. This is designated by the stillman as "the boiling of the still." The water soon decreases quite suddenly, and the stream at the receiving house slackens. The stillman then knows that all moisture has come off, when he says "the still has settled back." All this, however, will not take place when the crude is free from water.

The fires are now increased. As soon as the stream in the separating box has become clear and white, it is sampled and the gravity noted (in all this work the Baumé scale is used). The very first oil distillate, being colored and cloudy, is run into a separate tank to be rerun with a succeeding charge of crude. As soon as it has become clear and white and of proper gravity (75° to 95° Baumé), it is turned into the light naphtha tank by means of the stop-cock shown in d_1, Fig. 3. The stillman now observes the stream and from time to time determines the specific gravity. As soon as he arrives at the gravity at which he is to stop his light naphtha "cut," he closes this stop-cock and opens the one leading to the heavy naphtha or benzine tank. This cut is quite arbitrary, some refiners running all the naphthas into one tank. He continues as before and at the same time increases his fires slightly. Having arrived within a half degree of the gravity at which he is to begin the illuminating oil cut, he crowds his furnace to its full capacity.

11. In order to assist the distillation and produce a fine burning oil of sweet odor, the still is provided with a perforated steam pipe extending the full length of the still on the inside about 6 inches from the top. A very small quantity of steam is turned into the still, which helps to carry over the vapor more rapidly and prevents burning. When the stillman reaches the point at which the benzine is cut off, he directs the stream into the illuminating-oil tank.

This point is determined by local conditions, by laws regarding the burning point, and by commercial usage. The still is now kept running at full capacity until the stream reaches about 41° Baumé (this varies with the crude used), when it shows signs of color. The fires are slackened and the gravity will become lighter in the stream and the color will be preserved. This is the beginning of the so-called *cracking process.* The distillation is now continued into the high-test "water-white" distillate until a slight gas issue will be observed from the gas pipe outside the receiving house, which can usually be determined by several trial runs, and gauged by the stillman thereafter as so many hours after reaching 41° Baumé when the firing was reduced.

12. If the primary object of the refinery is the production of illuminating oils, one of two courses may be pursued: (1) The stream may be turned into a second-grade distillate tank and the oil run to tar of 19° to 21° Baumé. This mineral tar that remains in the still is permitted to cool to about 300° F. and transferred to a smaller still with extra-heavy bottoms and good internal bracings, to be again heated and distilled for paraffin and heavy paraffin oils. (2) It may be cooled after the first-grade oil is removed and transferred to a smaller still completely bricked in and having an elevated vapor outlet to carry out the cracking process, as previously explained. If, however, the crude oil is of such a nature as to permit the manufacture of good cylinder stock, a third course may be pursued after the first-grade illuminating oil has been removed, and that is the manufacture of wax oils and lubricating stocks.

13. As the average still attendant in a refinery is not a scientific man, it is necessary to give him exact directions as to the gravity at which to "cut" his oil in order to produce the grades required. This is sometimes quite difficult, on account of the great variety of requirements to which an American refinery is subjected. These cuts depend not alone on the crude oil, but more particularly on the market to which the salesman is catering.

These points are usually determined by making flash tests and fire tests of the different fractions and noting the quantity of distillate between each degree of gravity, in a run of the still to be used. After having arrived at a conclusion as to the grade of oil that it is advisable to produce, the stillman has simply to follow the directions as to firing and "cut" within the gravity limits prescribed.

14. Table I gives the results of a test made on a run of North Lima crude.

TABLE I

"Cut," Degrees Baumé	Distillate. Gallons	Flash Test
59–58	275	Flash below 59° F. in Tagliabue open cup
58–57	280	
57–56	275	
56–55	256	
55–54	300	60° F., Tagliabue open cup
54–53	285	73° F., Tagliabue open cup
53–52	290	75° F., Foster closed cup
52–51	315	93° F., Foster closed cup
51–50	375	95° F., Foster closed cup
50–49	360	109° F., Foster closed cup
49–48	400	112° F., Foster closed cup
48–47	310	122° F., Foster closed cup
47–46	335	135° F., Foster closed cup
46–45	298	144° F., Foster closed cup
45–44	306	158° F., Foster closed cup
44–43	280	163° F., Foster closed cup
43–42	295	172° F., Foster closed cup

Referring to Table I, the stream was allowed to reach 42° Baumé and the fires were permitted to burn lower in order to preserve the gravity and color. The distillation was continued in this way for 12 hours, the gravity fluctuating between 43° and 40° Baumé. All samples taken would

flash below 60° F. in the open cup. This was caused by the cracking of the oil. All flashes showed signs and gave the odor of sulphur. The flash test of this oil was readily raised to over 200° F. by steaming or blowing hot air through it.

15. Spray Condenser.—Another class of condenser is what is known as the spray condenser. In this condenser the oil is mixed directly with a stream of cold water as soon as it leaves the main vapor line. This insures quick condensation and the use of the entire value of the cold water. The simplest form to be constructed is to use a 4,500-gallon cylindrical car tank and set it on end. The vapor line should enter the center of the head on top and extend down through about 18 inches into the water in the tank. Entering the vapor line as it enters the tank is a perforated water pipe, which is fed from a constant supply of cold water. The oil is carried to the tail house through a 3-inch pipe from near the surface of the liquid in the tank, which is about 18 inches from the top. The water is drawn off at the bottom, thus insuring a perfect separation.

This condenser is found to be especially valuable in the distillation of Ohio crude. Many believe that the water removes considerable sulphur from the oil, which seems entirely probable, especially where alkaline waters are used. The principal advantage, however, is the speed to be obtained by this method, it being almost impossible to burn any of the vapors where a good supply of cold water can be had.

16. Cracking Process.—The cracking process, as it is technically known, is the process whereby heavy oils and residuum are converted into specifically lighter oils, having a lower boiling point. This makes them suitable for illuminating purposes. The process is both of scientific interest and technical value. Peckham claims that there is only 10 per cent. of high-test first-quality illuminating oil to be found existing as such in the crude oil; but as much as 70 per cent. is made from the oil. Much of this, however, will be of a low fire test. The hydrocarbons standing

between the kerosene and the lubricating oil, in gravity and boiling point, are of little value, and by this means may be converted into a salable article. Therefore, the proper manipulation of such a process is of great importance.

It is generally understood that the products of a fractional distillation, even in the laboratory, are by no means identical with the compounds existing in the crude petroleum. Professor Silliman, Jr., observed this fact in the irregularity of the boiling point in the distillates.

17. Thorpe and Young subjected solid paraffin to the action of heat under pressure, and thereby produced liquid hydrocarbons with but a slight loss as gas. A number of other chemists have conducted heavy petroleum through heated iron tubes and thereby produced lighter oils. The compounds produced have been found to belong to another series, viz., olefine, benzol, naphthalene, etc. This seems to convert the heavy American oil into the series most prominent in the Russian petroleum. The investigation of Professor Mabery in the distillation of California petroleum in a vacuum, which is just the reverse of the cracking process, has brought to light some new facts that may throw light on the compounds existing in the crude petroleum. As Mabery states, "none of the vacuum distillates nor the residue showed the slightest indication of decomposition; all portions had the natural odor of the constituents of the crude oil. From the lower fractions scarcely any paraffin could be separated."

18. In carrying out the cracking process, the principal object is to distil the oil at a higher boiling point than the normal. This object may be secured by having a large dome in which the vapor is partly condensed and made to fall back on the heated liquid; or, by elevating the vapor outlet, and instead of giving this line a fall, permit it to rise a very little before it reaches the condenser. A large number of arrangements for carrying out this process have been devised and patented, very few of which are in use today.

In most refineries little attention is paid to the matter, on account of the inferior quality of the oil produced by this means. Peckham lays stress on the following points, viz.: A pure, water-white oil of American origin is composed of the saturated-paraffin series, and on treating it with sulphuric acid the latter does not form compounds with the oil, but only with the parts which are foreign. Thus, after washing and neutralizing, nothing remains but the pure hydrocarbons.

In the "prime-white" oil, composed of the cracked oils, which is made up of olefines, the sulphuric acid finds compounds with which to combine, and on washing and neutralizing it will not be a pure hydrocarbon oil, some of the acid remaining in combination. Such an oil will readily darken in color, give off offensive fumes, and clog the wick, and cause endless trouble. In fact, here we have all the elements which produce complaint in the trade.

The only part of the process that is generally practiced is to reduce the fire and permit the exposed part of the still to carry out the partial condensation, and this falling back on the heated liquid may produce a closer fractionation if there is no decomposition. In most cases the oil remaining in the still is considered too valuable as a cylinder stock to be ruined in producing an inferior illuminating oil. In the distillation of the Ohio sulphur oils, however, this process has proved of great benefit.

The stocks remaining in the stills contain too much sulphur and other impurities to make a first-class lubricant without chemical treatment, and the breaking up of the heavy oils containing sulphur aids in their refining. Many patents have been taken out for the refining of Ohio oils, in which the salient point was really the breaking up of the sulphur compounds by a reheating of the vapor.

19. Distillation of Crude Oil for Lubricating Stocks. In a refinery aiming to make cylinder or other lubricating stocks from its crude oil there are some modifications necessary to the distillation described heretofore. The principles

adopted are such as to avoid anything approaching the cracking process. In fact, every effort is put forth to preserve the heavy part of the crude oil in the same form as it exists in nature. In order to carry out this process, the distillation is assisted by superheated steam. This causes the oil to distil at a lower temperature and the admixture of steam prevents scorching from the heated still walls. The still as described in Art. **3** is provided with two 2-inch perforated pipes as shown in Fig. 4 at *a*, *a*, lying close to the bottom of the still. These pipes are about

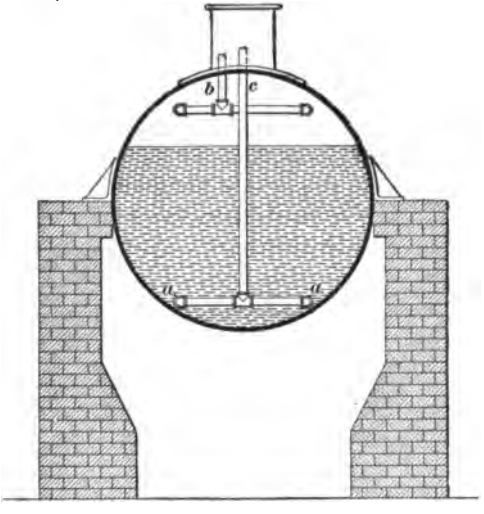


FIG. 4

1½ feet apart and reach throughout the entire length of the still. The perforations are made in two rows on opposite sides of the pipe with a $\frac{1}{16}$-inch drill. They are made so as to cause the holes to be turned downwards from a true horizontal position, thus causing the steam to strike the walls of the still and continually agitate the distilling liquid.

Just outside the still on the pipe *c* is placed a first-class Fairbanks globe valve. This valve is certain in its action and does not permit any steam to pass when closed. The steam before reaching the still may be passed through a

superheater, but if the still is close to the boiler house and a high-pressure steam is used, good results will be obtained without further superheating.

The still is charged and run as previously described with a little steam in the top line *b* until the point is reached where the first signs of gas begin to appear, showing decomposition of the heavy oil remaining in the still. Thus the crude has yielded its naphtha and first-grade burning oil in the usual manner.

In order to avoid even the slightest decomposition which will taint the lubricating stock, a trial run is made and the gravity of the oil in the still taken. All succeeding runs may be gauged by gravity instead of awaiting the signs of decomposition. In most grades of Pennsylvania crude oils, this point is close to 30° Baumé. When this point is reached, the fires are permitted to burn low. The moisture is all blown from the steam line entering the still through *c* by means of a small valve attached close to the valve on the outside of the still.

20. As soon as the steam has a blue appearance the valve on the line *c* is opened about $\frac{1}{16}$ turn. This will cause considerable commotion, and the stream at the receiving house will increase rapidly at once; but after a very few moments it will become quieted somewhat, when the valve is further opened to $\frac{1}{8}$ turn. The line conducting the top steam to the still is now shut off and the other valve opened wider from time to time until it is opened to its full capacity. If all lines leaving the receiving house have sufficient fall, the operation will require but a very short time. The firing may now be resumed and so regulated as to have a stream in the separating box, in which the oil and the water from the condensed steam stand about in equal proportion. If a superheated steam of 450° F. is at hand, it is well to keep the fires very low at the end. The fire test and viscosity of the oil depend entirely on the workings of the steam.

In order to distribute the steam throughout the still to better advantage, some have found it more satisfactory to

have the bottom pipe constructed with a number of cross-arms, which are bent to the curve of the bottom of the still. By testing the oil in the still, it is seen that the fire test and viscosity rise as the gravity falls. The stillman again uses his hydrometer as his guide, and produces an oil of the required fire test and viscosity. When run with care, it seldom varies more than 2° F. in fire test from the same gravity of a previous run. The cylinder stocks used are either 600°, 650°, or 700° F. fire test. Some stocks for special filtering run as low as 500° F. fire test.

21. The distillate coming off after the introduction of the bottom steam soon becomes colored, and contains a large percentage of solid paraffin; the first part of it may be cut into a separate tank of 34° to 36° Baumé oil to be used in the compounding of miners' lamp oil, while the remainder, known as wax oil, is run into one tank and sent to the paraffin works. Having reached the required gravity in the still, the fires are drawn and the still pumped out as soon as advisable. If it is not pumped out very soon, the good accomplished may readily be undone. The vapors remaining in the still and settling back on the oil will give the latter a bad odor and taste, or if the steam is left in the oil after the fires are drawn, the moisture, which partially condenses on cooling, will make it unmarketable. Hence, to save the oil, it must be removed as soon as possible. In pumping out hot oil like this, the bottom steam is shut off and steam introduced in the upper line, so as to prevent any vacuum being formed in the still, on account of cooling and the removal of the oil. Destructive explosions have resulted from a neglect of this precaution, owing to the fact that the hot vapors of petroleum remaining in the still when mixed with air coming through the worm will ignite spontaneously.

This oil is marketed as steam-refined cylinder and grease stocks of various grades. When properly refined, all light oils removed, and no decomposition has taken place, the oil is perfectly tasteless and odorless. Much care is required in

every part of a distillation of this kind. One of the most important features, however, is a constant and unchanging steam pressure and regular firing, so as to keep the still running an unvarying stream.

CHEMICAL TREATMENT OF THE DISTILLATE FOR BURNING OILS

22. Introductory.—The chemical treatment of the distillate derived from petroleum has an interesting history, the rank odor being one of the sources of trouble at the very beginning of its use. The first shipment of oil arrived in New York in 1857, and its strong odor was the cause of much complaint. The oil, besides having a strong odor, was of a yellow to dark-brown color and with age became darker. Its burning quality was very poor on account of the formation of a crust on the wick, which destroyed the capillary action of the fibers. In addition to these evils, the lamp emitted a very offensive smelling gas while burning. In spite of all these objections, oil was in such demand that the price rose to $2 per gallon.

In order to overcome these difficulties, a zinc-lined tank was constructed in which to chemically treat the oil. The tank was partially filled with oil and a hot solution of caustic soda added and the whole agitated with a wooden paddle. This was probably the first agitator used in America. The treatment was satisfactory in so far as it removed the smell, and the color also became lighter, but the oil soon turned dark again and deposited a solid crust upon the wick after burning a short time. It was next tried to free the oil from remaining lye by washing with dilute hydrochloric acid, but the color and burning quality remained the same.

23. Treatment With Sulphuric Acid and Caustic Lye.—Eichler, of Baku, is stated to have been the first to introduce, in Russia, the use of sulphuric acid and subsequent treatment with lye, and thus created a method which is in

general use today. The action of sulphuric acid on the distillate has not yet been fully explained, but it appears to consist in the removal of aromatic hydrocarbons, petroleum acids, phenols, tarry products, etc. The manner in which these foreign products are removed is by no means the same in each case; some are partially destroyed or dissolved and changed to sulphonic-acid combinations, while others remain almost entirely unchanged and are removed only by the subsequent treatment with lye. The tarry matter in the distillate is dissolved by the sulphuric acid and imparts to it the dark color. The phenols, sulphonic acids, and the remaining sulphuric acid are neutralized and removed by the caustic soda. The acid is especially instrumental in removing the oxidized compounds of the distillate; in fact, by continuing the treatment all oxygen can be removed.

The action of the sulphuric acid is more complicated than appears at first sight. The evolution of sulphurous acid, which always accompanies a treatment, is not accounted for in the reactions given above. Its appearance can only be accounted for by the reduction of part of the sulphuric acid; but as to the compounds causing this reduction, nothing is known. That the acid actually attacks the compounds of the oil is true, since each succeeding acid will continue the action, evolve gas, and take on color. Its action does not seem to be limited. The longer the acid remains in contact with the distillate, the deeper will become the color of the latter. In fact, a certain color of the distillate in the treatment is one of the limits that the treater must not pass in order to produce the best results.

24. The quantity of acid required and the length of time during which the treatment is continued depend on the purity of the distillate, and this in turn depends on the composition of the crude oil and the manner in which the distillation was conducted. Cracked oils require more than the uncracked oils, and the Ohio sulphur oils more than the Pennsylvania oils. Usually the amount of acid required increases in proportion to the density of the distillate.

25. Agitator. — In order to secure prompt reaction between the oil and chemicals, they are intimately mixed by some mechanical appliance in a tank called the agitator. Those commonly used for the treatment of light oils consist of upright cylindrical vessels, with conical bottoms. They rest upon strong brick or stone piers, or upon an iron construction, as shown in Fig. 5. When located in a closed



FIG. 5

building, they are open at the top, but if erected in the open air, they are provided with a light iron hood, fitted with trap doors, to protect the oil from rain and from the direct rays of the sun, and still allow of a free circulation of the air.

They are constructed similar to all other tanks about a refinery, except that they have a lead lining to protect the

iron from the action of the dilute acid formed when there is moisture in the oil, and also at the beginning of the wash with water. The thickness of the sheet lead is usually from $\frac{3}{16}$ to $\frac{1}{4}$ inch, but is sometimes as much as $\frac{5}{16}$ inch. The lead plates are laid on the sides of the agitator and lapped over the edges and soldered together, so that, as it were, they hang in the apparatus. It is customary to construct the agitators for light oils with a greater depth than width, because a more intimate mixture of oil and chemicals can be effected in a tall, narrow vessel than in a flat one. The cone is likewise constructed with steep walls, its depth being about two-fifths that of the diameter of the upper part. This allows a rapid settlement of chemicals, which is particularly important in the acid treatment.

Nearly all agitation is now carried out by means of compressed air, the air line entering the agitator at the top, and passing down to the lowest point of the cone. In addition to a pipe by which the agitator is charged with oil, it has separate pipes for acid, alkali, and water. The chemicals are forced in by means of compressed air. The bottom of the agitator is provided with a three-way stop-cock, with bores of large diameter to prevent their choking up by thick, tarry sludge, or it may be provided with any other suitable stop-cocks and lines, as at *a*, Fig. 5, for discharging the acid, lye, water, and oil into their proper receptacles.

26. In making 150° water-white oil from Pennsylvania crude, the stillman uses the "cut" from about 58° to 40° Baumé. This will produce a distillate of light, lemon-yellow color and about 49° Baumé and about 110° to 115° F. fire test. In order to raise the fire test, the distillate is pumped to the steam still similar to the one used in rerunning naphtha. It is here steamed very carefully until all of the light oils have been removed and the distillate has the required flash point. It is now pumped to the agitator and cold water pumped through it until it has cooled to about 60° F. After standing some 5 or 6 hours, all the moisture will have settled out and is drawn off.

In oil of this grade it is customary to use about 6 pounds of sulphuric acid of 66° Baumé per barrel of oil. This is usually added in two portions, a little less than one-half being used the first time. This portion of the acid being drawn off into an air-tight drum and connected with the air compressor, the air blower is started and the oil put in agitation. The compressed air is then turned into the drum and the acid discharged into the oil. The agitation is now continued regularly and at such a speed as to keep everything intimately mixed. The attendent observes his oil very closely during the entire process. Samples are from time to time taken in a test bottle and the action of the acid judged by the manner in which it settles. In a very short time the acid is seen to settle in small, elongated drops, which adhere to the sides of the test bottle. Shortly after this the acid forms in small beads, which become larger as the process continues, and settle rapidly to the bottom of the flask. This is the point the treater desires to reach. The acid has accomplished its work and the blower is shut off. The operation thus far usually requires from 30 to 45 minutes. Should the temperature rise more than 2° or 3° F. before this point is reached, it is well to discontinue the operation, as a rise in temperature always indicates too violent action on the part of the sulphuric acid. This may be caused by moisture in the oil, or by too much moisture in the atmosphere.

27. After the acid has been permitted to settle for from 45 minutes to 1 hour, the sludge or spent acid is drawn off. The second or larger portion of the acid is now added in the same way as was the first and agitated about the same length of time. The treater will now observe that the oil assumes a cherry-red color and violet florescence, and that the acid settles somewhat slower; when, however, it has reached its limit, it quickly clears in the neck of the bottle, leaving the oil white. The acid treatment may be considered finished as soon as a sample of the distillate forms a milk-white emulsion when mixed with dilute lye; a series of

these tests will soon show the practiced eye when the action is complete. Here, again, the same care must be taken as to a rise in temperature. It is well for the treater to wash a small sample to see what color it will produce.

The oil having been permitted to settle for at least 2 hours, the sludge acid is drawn off. The time of settlement depends very much on outward conditions and the distillate treated. The treater must keep a watchful eye on his oil during this time, and should he at any time observe any change in the color towards the violet, it will be well for him to wash a sample and note the results. The sludge should be drawn off every 15 minutes after the first half hour, for final results rest on the purity of the oil at this point. It is to be observed here that more complications arise from the use of too much acid than too little.

After all due precaution has been taken in the foregoing operations, the oil is washed with cold water. The water is pumped on to the surface of the oil in a fine spray and drawn off at the bottom. This is accomplished by means of a rose nozzle or perforated pipe running around the top of the agitator. It is well to wash down the sides of the agitator as soon as possible, for all traces of sludge must be removed quickly if a first-class oil is desired. This wash continues with intermittent agitation until all traces of acid have been removed and the water passes through the oil perfectly clear, and shows no signs of acid with litmus solution. The oil is now permitted to settle for some time (about 30 minutes) and the water drawn off. It is again agitated and 25 pounds of caustic soda, dissolved in water to about 6° Baumé strength, added in a small stream. This agitation is continued for about 20 minutes, when the oil should be thoroughly alkaline.

The oil is now permitted to settle for from 5 to 6 hours, when the alkali and soaps settle to the bottom and are drawn off. It is to be observed here that an undue amount of soaps and slow settling are signs of too much or too long acidification. The oil is now thoroughly washed with warm water of about 90° F. This wash is continued until the

water flowing from the oil shows no signs of alkali on being tested with a red litmus solution. The oil is now transferred to large flat tanks, known as settling tanks or bleachers, where the remaining soaps and moisture are settled out. These tanks were formerly all so arranged as to enable the treater to permit the direct rays of the sun to act on the oil and bleach it.

28. The best illuminating oils are not bleached at present; they are usually settled by means of the steam wash. In this process, a perforated steam coil is placed in the bottom of the bleacher and the oil steamed until it reaches a temperature of 110° F., when it will settle out clear and bright within an hour. The oil is now ready for the market and may be shipped directly or placed in the storage tanks awaiting orders. Much has been done to produce marketable oils from the heavier and poorer stocks; but nothing has been found to equal a light cut of Pennsylvania distillate after a careful treatment as described.

In case of a treatment of cracked oils, which contain much more oxygen than the regular distillate, the acid process is not adequate to remove all foreign compounds with two applications. It is customary to use a preliminary treatment of concentrated lye of 25° to 30° Baumé. This removes the oxygen compounds, much tar and foreign matter, and gives the acid more opportunity for good action. Even when oils of good color and odor are produced from such distillates, they readily take up oxygen and deteriorate in color, odor, and burning quality.

29. Treatment of Oils Containing Sulphur.—Among the almost endless patented processes for removing sulphur from Ohio and Canadian oils, there is only one, the treatment with sodium plumbate, which has received the attention of all refiners. Although it has been discarded by many, and other methods used in its stead for a time, it is still more generally employed today than any other process. The only reason that refiners fail to make an absolutely

acceptable oil by its use is the lack of intelligent and painstaking treaters. It is a delicate chemical reaction and should receive the attention of a perfectly trained eye and hand.

30. Sodium-Plumbate Treatment.—The oil is distilled and treated in the same manner as the Pennsylvania distillate just described, until the alkali treatment has been finished. Now, instead of washing the oil with hot water, it is heated with an open steam coil to about 110° F. and agitated with a hot solution (200° F.) of sodium plumbate, prepared as follows: Make up about 10 barrels of caustic-soda solution of 22° Baumé and dissolve in this 400 pounds of the best litharge. This is heated with steam almost to the boiling point. About 5 barrels of this is pumped into the hot oil and agitated for 20 to 30 minutes. The color changes to a dark brown, and at times to almost black. The treater tests his oil from time to time to see if it will stand the "doctor." To make this test, a sample of the oil is settled with a little flowers of sulphur and the clear oil decanted into a testing bottle. Now there is added the "doctor," a concentrated solution of sodium plumbate. The oil should not change color. If it turns a faint-yellow or dark-brown color, the lead-and-lye treatment is not complete. This state of affairs should take place quickly, and should it not stand the above test within 10 or 15 minutes after the beginning of this treatment, it is well to add more of the lead-and-lye solution immediately.

As soon as the oil stands the "doctor," shut off the air and permit it to settle. Within half an hour the greater part of the lead-and-lye solution can be drawn off and run back to the tank with the part remaining. As will be noticed above, we have prepared a large amount of chemicals, but most of these have been returned and can be used again. In the average treatment, about $\frac{1}{4}$ pound of litharge and $\frac{1}{4}$ pound of lye per barrel of oil are used. If the oil has been well treated, it will now settle out almost clear in the agitator. The more lye and moisture that can be settled

out before proceeding, the better will the oil be when finished.

Some have washed it with water at this stage, which, however, necessitates a thorough settlement or reheating to remove the moisture.

The oil is permitted to cool to about 90° F. and the lead and lye drawn off as close as possible. It is again put in agitation and about $\frac{1}{16}$ pound of flowers of sulphur per barrel added in small portions. The oil is again sampled from time to time, and as soon as the black, flocculent particles of lead sulphide leave the oil clear in the neck of the bottle, it is finished. As flowers of sulphur is soluble in the oil, too much sulphur will destroy the good that has been accomplished in the lead-and-lye treatment.

The oil is now permitted to settle for 5 or 6 hours, when all the lead sulphide settles to the bottom, leaving the oil clear and sparkling. This sediment is composed of lead sulphide and a heavy tarry substance, doubtless compounds of the hydrocarbons, which have combined with the lead, lye, and sulphur. It forms a slight crust over its surface, which should not be disturbed again. It is necessary to draw off the oil by means of a swing line connected with the agitator above the cone, so as to leave all this sediment undisturbed. It is impossible to make an oil that will not clog the wick and leave heavy incrustations, if the black sediment is permitted to get up through the oil again after the first settlement. The sediment seems to act as a filter to the oil.

31. Grading of Burning Oils According to Color. The burning oils chiefly manufactured in the United States, both from Pennsylvania and Ohio crude, are usually designated by the fire test and color. The water-white oil of 150° F. fire test has been considered the standard oil in America for some time. Now it is necessary to call for a 48° Baumé gravity, 150° water-white oil, in order to obtain the high-grade oil, since we find on the market 45°, 46°, 47°, 48°, and 49° Baumé, 150° water-white oils. Besides the colorless water-white oils, there are all tests and gravities of

prime white, a faint yellow; and *standard white*, a pronounced yellow. All these grades may be 110° oil of 110° F. fire test; 120° oil of 120° F. fire test; 150° oil of 150° F. fire test; and 175° oil of 175° F. fire test. Besides these brands, the gravity is looked upon as more prominent in the grading than formerly, and is receiving its just recognition in the production of a good, light oil.

The 175° oil should be of a water-white color and 45½° Baumé gravity. It is frequently sold under the brand of *headlight oil*, but all so-called headlight oil is by no means 175° oil. Its principal use is in the lamps of the headlights of locomotives. The United States Government frequently contracts for oil of 135° F. flash test, to be used as a safety illuminant for general purposes. Besides these grades, there is manufactured to a comparatively small extent a burning oil of 300° F. fire test, and of 36° to 38° Baumé gravity. Formerly, a standard white was sufficient, but at present it is required to be water white. This oil was first introduced by the Downer Kerosene Company, of Boston, under the name of *mineral sperm*. At present it is sold under the names mineral colza oil, mineral sperm oil, mineral seal oil, coach oil, and 300° oil. It is used for an illuminant in railway coaches, lighthouses, and for several other purposes where readily ignitible oils are objectionable.

THE REFINING OF NAPHTHA

32. Introductory.—If the crude oil is rich in the very lightest hydrocarbons, they are separated into various cuts as they come from the crude still. These crude naphthas, or benzines, as they are frequently called, can seldom be used as such, it being necessary to deodorize and refractionate them. Some of the lightest products are sometimes taken as they come from the crude still, such as 90°, 88°, and 80° Baumé gasoline. These products are used in the air gas machines for the manufacture of gas for lighting or heating, by passing air through gasoline. This mode of working in the refinery is not profitable to the refiner or the user of the

gasoline, because the products thus obtained are less valuable, the carrying over of heavy products being unavoidable in the regular crude distillation. The best method is to make not more than two cuts at the crude still, viz., the light and the heavy naphtha, and refractionate these for high-grade gasolines.

33. Chemical Treatment of Naphtha.—The crude naphtha is pumped to the agitator to receive a chemical treatment similar to that which is applied to the oil distillates. For light naphtha, about 4 pounds of sulphuric acid of 66° Baumé per barrel is used, and all is added at once, unless there is danger of moisture, when it is well to use about one-fifth of the entire amount to remove the water, after which the agitation with the principal acid is continued for from 15 to 30 minutes, this time depending largely on the temperature of the atmosphere. If it is warm, the acid will take hold of the tarry particles very readily and the time is cut short in order to avoid loss by evaporation.

When the naphtha has been sufficiently agitated, the air blower is shut off and the heavier acid is permitted to settle for at least 1 hour, and even longer in cold weather. The sludge acid containing the particles of tar is drawn off after standing half an hour. After this it is drawn off about every 15 minutes, and when found to be free from sludge after standing 15 minutes, the naphtha is ready to receive the caustic treatment. Having dissolved about 15 pounds of pure caustic soda in water to a strength of 8° Baumé, the naphtha is again agitated and this solution forced into it. The agitation is continued for about 15 minutes and the mixture is then permitted to settle. After drawing off this caustic and dissolved soaps, a sample of naphtha taken from the top of the agitator and tested with hot water and a drop of litmus solution *must be free* from acid. The naphtha in this condition is called *deodorized naphtha*, since it has lost the penetrating odor which it had as a distillate.

It will be observed that the wash with water, after the acid treatment, has been omitted in the naphtha treatment.

The acid is so much heavier than the naphtha that it settles out very quickly and quite completely; the aim in the treatment is to shorten the process in order to save the naphtha, which is very volatile, to avoid a loss which may easily amount to several per cent.

34. Distillation of Naphtha.—The deodorized naphtha is now pumped, or, better, permitted to run by gravity, to the gasoline still, sometimes called the *steam still.* This is commonly a cylindrical still provided with a steam coil, similar to the bottom coil described under the cylinder stock distillation, only it is not placed on the bottom, but about 6 inches from it. This gives some space underneath the steam coil for the collection of water, which will not be disturbed during the distillation. The still is built similar to the other still described, with the exception that it is not necessary to make any distinction between the weight of the sheets used on the top or bottom, since fire is not used in this work. The still is placed on a good brick foundation and entirely covered over with a brick jacket.

35. Naphtha Still.—Many small stills have been devised that were capable of close fractionation, but in a gasoline works, where it is necessary to distil large quantities each day, a still constructed on the same principles as the one shown in Fig. 6 is used. The principal features of this still are a vapor line *a*, which has a high raise before it reaches the condenser, keeping back all particles of liquid that may have been carried along with the vapor, and what is known as a *back trap* for close fractionation.

The coil in the condenser *A* is attached at its lower end to a 6-inch pipe (the back trap), from which the line *c* (the back-trap line) leads to the bottom of the still. The line *b* connects with the condenser *B*.

As soon as the deodorized naphtha has been heated to the distilling point by means of the steam, the vapor gradually rises in the line *a* and enters the condenser *A*. It traverses the coil and a large fraction of the heavy gas will be condensed and return to the still by means of the back-trap

line *c*; but the lighter gases, which are not so readily condensed, will rise and enter the condenser *B* through the line *b*, where they traverse a much longer condenser and become a liquid. Here the coldest water is absolutely necessary to prevent loss; and even then some gases will pass off through the line *d*. These may be caused to pass through

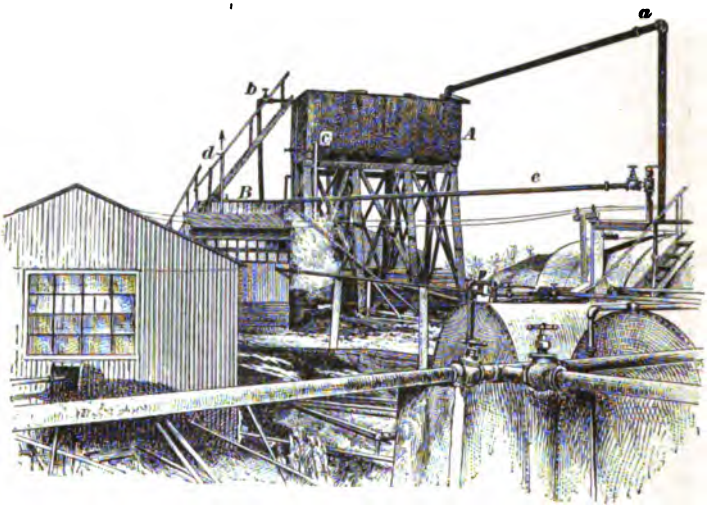


FIG. 6

a coil surrounded with a freezing mixture composed of ice and salt in order to condense the very lightest product, the *cymogene*. In this manner one is enabled to make a very close separation of light and heavy hydrocarbons. If 80°, 88°, or 90° Baumé gasoline is wanted, it may be separated here to a better advantage to both consumer and refiner than at the crude still.

36. In order to make the best products, the stillman must make his cuts by *weighing* (i. e., taking the Baumé density) both the tank and the stream. If the steam can be kept at a constant pressure, the still can be so arranged as to run practically automatically. Mr. Merrill, of the Downer Company, first obtained rhigolene, which has a

density of 94° Baumé and a boiling point of 65° F. Cymogene may be obtained by vaporizing some of the very light products, as rhigolene, and condensing them by a freezing mixture. It has a density of 110° Baumé and boils at 32° F. These products are used in surgery as local anesthetics.

Some of the more common products manufactured are stove gasoline, from 68° to 74° Baumé, and a heavier product of 63° Baumé, known as deodorized benzine. Since the introduction of stoves whereby the former article can be used for cooking purposes, it has become one of the most important articles of the refinery. The deodorized benzine is used by paint manufacturers in the preparation of paints, enamels, and varnishes, and by pharmacists as benzine for cleaning purposes. This benzine should be free from oil and leave no sign of grease on a piece of white paper or silk.

Formerly much of the naphtha was fractionated and then treated; this has, however, given way entirely to the process given above. The redistillation after treatment leaves all chemicals behind and there is produced a pure hydrocarbon, perfectly safe to be used for any purpose for which it may be required.

37. The heavy naphtha, from 58° to 62° Baumé gravity, coming from the crude still, is frequently marketed without treatment as crude gas naphtha. It finds its use in such a case to enrich coal gas. If refined, it is treated in a similar manner to the other naphthas and rerun in the gasoline still. In rerunning this, the upper condenser is not used and the vapor is permitted to pass through the line *e* directly to the condenser *B*.

The whole naphtha is run into one grade, the 63° deodorized for paints, varnishes, etc. This still is used in the same way when raising the fire test of the distillates.

The naphtha from the sulphur oils is treated in a similar manner to those from the Pennsylvania crude, excepting the additional use of a small percentage of sodium plumbate. In the light naphtha this is found to settle out without the

use of sulphur; but in the heavy naphtha it also requires some flowers of sulphur to clarify it similar to the oil treatment.

38. Tables II and III, taken from Redwood's "Petroleum," Vol. I, give the products manufactured by two different refineries from the light oils.

TABLE II

Product	Density. Degrees Baumé	Specific Gravity	Use of Product
Rhigolene or cymogene	90	.636	For surgical purposes as a local anesthetic.
Gasoline	88–86	.642–.648	For air gas machines.
Boulevard gas fluid.	76	.682	For street naphtha lamps.
Prime city naphtha (benzoline)	73–68	.692–.709	For sponge lamps, etc.
Benzine	62	.731	For oilcloth and varnish making.

TABLE III

Product		Density. Degrees Baumé	Specific Gravity
Petroleum ether.	1. Cymogene	108	.590
	2. Rhigolene	94–92	.625–.631
	3. Gasoline	90–80	.635–.668
Petroleum spirit.	4. C naphtha (benzine naphtha).	76–70	.682–.702
	5. B naphtha	66–65	.716–.720
	6. A naphtha (benzine)	59–58	.743–.747

39. All the products in the tables above sink into insignificance when compared with the stove gasoline, which in many cases consumes everything, from the very lightest to the heaviest, its demand having far outstripped the fondest hopes of many of our wide-awake refiners.

In speaking of the utilization of the light products of Russian petroleum, Crew says: "The light oils evaporated during the first distillation are allowed in most instances to run into the sea, but lately certain refiners have commenced to work them into gasoline and benzine, and, though the market for these products in Russia is limited, they yield a reasonable profit." This same thing can be said of the American light oils prior to about the year 1885. Then the naphtha was run into streams or used as fuel under the stills as a waste product.

Since it was first refined it has gradually worked its way to the favor of the public until, during the summer of 1898, all grades, from the very lightest to the heaviest, commanded a higher price than the best grade of kerosene. From being a by-product in the petroleum refinery, it has taken first place. Use has been found for it for cooking and heating, for whatever purpose gas could be used, and, consequently, it has brought comfort to people of the rural districts.

THE PARAFFIN INDUSTRY

40. Introductory.—Notwithstanding the fact that paraffin had been known for a long time, its manufacture on a large scale is of a comparatively recent date. It was known to exist as ozokerite, or earth wax, in Europe and various parts of America, but the discovery that it is a product of distillation of several organic bodies belongs to Karl Reichenbach, who gave it the name paraffin, from *parum* and *affinis*, because it appeared to be wholly destitute of chemical affinity. Its commercial value is based on the work of Seligue, of France, who manufactured it from bituminous shale. The production from this source has grown to enormous proportions in Europe and had taken a fair step in America when the more abundant and cheaper crude material, petroleum, took its place. In this country it is now entirely obtained as a by-product in the petroleum distillation, and this, like many of the other by-products of

petroleum, has grown to such proportions as to become almost the principal product in certain petroleum distillations. It does not possess a constant composition; but, like all the other products of petroleum, it is a mixture of a number of hydrocarbon compounds.

In treating the subject from the standpoint of the refiner, the preparation of lubricating oils and the manufacture of paraffin wax run so closely together that in some points at least they touch, and must be treated as if they belonged to one subject. The manufacture of either or both begins with the distillation of the residuum coming from the illuminating oil distillation or the crystallization of the wax oils coming from the lubricating-stock distillation.

41. Distillation of Oil Residues.—The mineral tar or residuum is transferred after becoming entirely cold, or is reheated and then transferred into stills of about 300-barrel capacity. In the largest works much larger stills are also used. These stills have especially heavy steel bottoms and good internal bracing. The outlet, or vapor, line is large and is divided, by means of a U-shaped arrangement of the lines at the condenser, into two parts, as shown in Fig. 7. The one line *a* remains on a level with the vapor line, passes around the condenser, forming an air condenser of from 80 to 100 feet, and finally enters the condenser box and passes to the receiving house through a separate line;

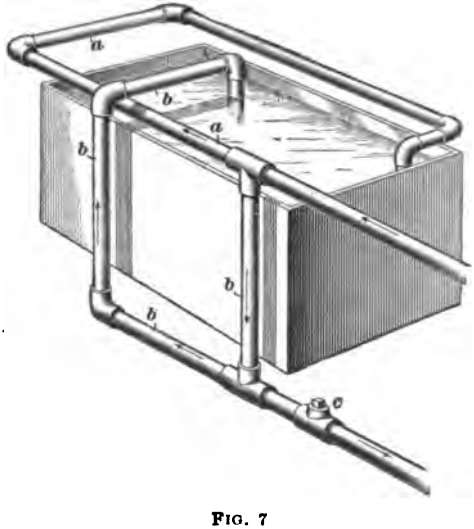


FIG. 7

the other line *b* makes a drop of about 5 feet from a **T** in the vapor line, and forming a **U**, rises again, enters the water box, traverses it several times, and thence passes to the receiving house. Thus, the still has two separate lines that carry off distillate at the same time. The water condenser is small and not kept too cool.

The still is heated gradually. If the tar has been chilled it begins by reporting with a bumping sound; this will increase in rapidity, but should not increase in force; if it does, it is a sign of moisture. A regular humming and crackling sound indicates boiling and no danger from moisture. This boiling may require 3 or 4 hours, according to the condition of the tar used. The fires are now crowded to their utmost in order to distil without cracking. The first distillates coming from both air and water condenser lines are run into a separate tank, to be rerun or used as a heavy gas oil. As soon as the oil coming from the water condenser line is clear and usually about 36° Baumé, it is turned into the paraffin distillate tank. The air line will run oil which is quite light and contains but little wax, and continues to run to the heavy gas oil, until near the end of the run, when it is found to contain wax and is turned into the same tank as the stream from *b*. Thus the stillman is enabled to separate the light oils from the heavy oils without refractionation.

The paraffin distillate is made in one long cut. The only point to be observed is to avoid too much color. The bottom of the still assumes a cherry-red color and the distillate becomes heavier and more waxy until the latter end of the run, when it is almost a pure wax. This is known as the *wax tailings*, and is permitted to run directly to a separate tank, by opening the stop-cock *c* on the vapor line. The whole charge of 240 barrels may be run off in from 8 to 12 hours. Since the still is run dry in order to get all oil and make a good coke, the stillman must be experienced in knowing just when to shut it down and not overtax the bottom, which with care should make from thirty to forty runs. The coke from the tar still is a valuable by-product, being

used in the manufacture of electric supplies, artist's carbons, etc.

42. The other source of paraffin in a petroleum paraffin works, as already mentioned, is the wax distillate coming from the steam distillation in the manufacture of lubricating stocks. This wax oil is again placed in the still and rerun without the use of steam in the bottom of the still. This is what is known as the crystallization of the wax oil. Much has been said for and against the idea of paraffin existing in the crude oil in an amorphous state. However this may be, it is a known fact to the refiner that unless the oil be distilled at a high temperature the paraffin will have a vaseline consistency and stick to the disks and filter-press plates; whereas the crystallized wax oils press easily and the wax comes off in hard and firm cakes. The cut from the crystallization of the wax oil is the same as the first making of a distillate of 30° to 34° Baumé. This, however, varies, according to the peculiar views of the refiner and the wants of his customers.

43. Treatment of Paraffin and Wax Distillates.—The paraffin and wax distillates are pumped to the paraffin agitator (see lubricating agitator described later). This is constructed on the same general principles as the other agitators described with the exception of a special arrangement for heating its contents and keeping the dense and easily congealed oil in a liquid condition. No more heat should be employed than is actually necessary to keep the contents in a fluid state and the paraffin entirely melted. The same general principles of chemical treatment followed in the case of the illuminating oil are observed here. The action of the acid on the oil is very energetic and is accompanied by the disengagement of large volumes of vapors containing sulphur dioxide. For drawing off the heavy acid sludge coming from this treatment, exit pipes and stopcocks of large internal diameter are required. The sludge on standing becomes quite solid, and on being neutralized

with lime forms an asphaltum which is excellent for roofing and light paving.

The acid treatment is followed by the usual water and alkali wash. This treatment being complete, the oil is transferred to a shallow tank provided with a steam coil, in order to keep the wax from congealing, and to permit the settlement of the water. This purifies the paraffin, removing all coke and petroleum acids, which are ruinous to the canvas of the filter presses.

44. Chilling.—The oil is next transferred to another shallow tank, where it is subjected to the chilling process. This was formerly carried out by means of a freezing mixture, composed of broken ice and salt placed about the cask containing the oil, similar to the arrangements in an ice-cream freezer. At present, ammonia ice machines are employed entirely for this purpose.

One of the methods in applying the cold produced by the evaporation of the liquid ammonia is to lead the ammonia pipes, together with a set of pipes to convey the oil to the filter press, through a vat containing a solution of calcium chloride. The evaporation of the liquid ammonia in the pipes, brought about by means of a vacuum pump, chills the solution of calcium chloride, which in turn cools the oil being pumped through the other coil, and thus causes the paraffin to crystallize. The wax oil may enter the vat at 80° to 90° F. and leave it at 10° or 15° F.

45. Pressing.—The first pressing is usually carried out at about 40° F. in a filter press (see Fig. 8). The chilled paraffin oil is placed in canvas bags and arranged in the filter press between perforated iron disks. The whole is subjected to a gradually increasing pressure, which forces out the liquid oil and leaves the solid paraffin behind. Unless this operation be skilfully conducted, enough crystallized paraffin may be forced through the canvas to interfere materially with the cold test of the oil.

These cakes of paraffin are melted and the fluid paraffin filtered through bone-black filters to remove the coloring

matter contained in it. It is again chilled and placed in the hydraulic filter press, which is similar to the first, in which it is subjected to a much greater pressure at a higher temperature than before. It is pressed for from 5 to 8 hours at 70° F. and at a pressure of 300 pounds to the square inch. This produces a wax melting at from 117° to about 120° F. The melting point varies somewhat with the gravity of the oil brought to the presses. The cakes thus formed are removed and ground up into small scales. This scale when not treated with bone black is what is known as *crude scale*, and much of it is sold in this form to be worked up for special purposes.

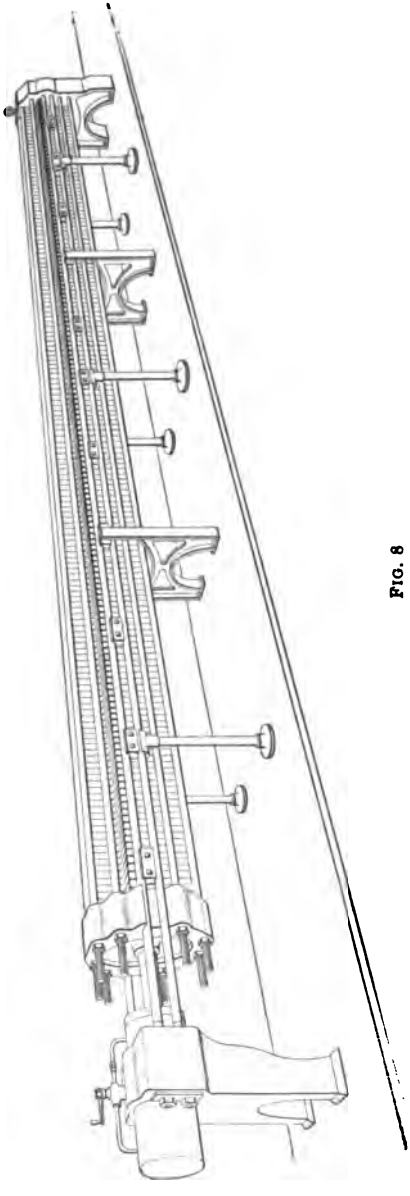


FIG. 8

The refiner making refined wax grinds the crude scale with benzine and by gently heating dissolves the wax. This is again cooled and subjected to hydraulic pressure. This is known as the *recrystallizing process*. The paraffin thus obtained is in large

crystals, perfectly white and transparent, and has a somewhat higher melting point. Where there are no particular melting-point requirements this wax is used exclusively. In order to meet the requirements of higher melting points and perfect freedom from oil, the paraffin is put through the *sweating process.*

46. Sweating Process. — The recrystallized wax is melted and run into shallow pans 3 inches deep, 4 to 5 inches wide, and about 24 inches long. It is chilled and these cakes placed in a small room surrounded by steam coils. The cakes are placed on racks having perforated bottoms, underneath which are tin troughs leading to a common receptacle. This room is heated to the temperature of the melting point that the wax is required to have and kept at that temperature for about 12 hours, when all the oil and low-melting paraffin will have dripped out of the cakes, which come out perfectly honeycombed, but of the desired melting point. The American paraffin is usually sold at the melting points of 125°, 128°, and 135° F., known as C, B, and A paraffin, respectively.

Since paraffin has been produced in such large quantities from the American petroleum, the large manufacturers have made especial efforts to introduce it into general use. By their persistent efforts in informing the public and placing it on the market in convenient forms, it is steadily but surely becoming a household article of inestimable value. The paraffin candle has completely superseded all other forms of illumination along this line. It is also taking the place of sealing wax in the preservation of jellies and fruits.

THE PRODUCTION AND MANUFACTURE OF LUBRICATING OILS

47. Introductory. — The pecuniary and beneficial advantages accruing to modern civilization from the introduction of the heavy products of petroleum for the lubrication of machinery are hardly second to those resulting from the introduction of the cheap and beautiful light furnished

by its illuminating portion. It seems strange that the second oil well in Pennsylvania should produce a petroleum so different from that found at the Drake well as to turn the attention of the promoters of the new field towards an entirely different industry. Such was the case: light and lubrication presented to the world simultaneously, one on Oil Creek, the other on French Creek, and both in a region comparatively isolated.

James Evans, a blacksmith at Franklin, drilled a hole in his water well, which had shown signs of oil at each recurring dry season, and at 72 feet struck a crevice. By means of a hand pump he succeeded in pumping up 25 barrels a day; and afterwards with steam power 70 barrels per day. It was a heavy oil, about 30° Baumé, free from grit and quite smooth. For some time the heavy oil was used principally in its natural state. By degrees, improvements were introduced and articles of a very high grade were produced by refining.

Joseph C. Sibley was the first to conceive the idea of manufacturing an oil from petroleum, to be used in steam cylinders. To-day engineers are enabled to carry higher steam pressure and operate with much less wear on the steam valves through its use.

Since the time that the Franklin oil was recognized to be of particular value as a lubricating oil, producers have been keen to examine carefully all heavy oils that might be found in any of their wells. Few, however, have found anything equal to that at Franklin. A field of considerable importance has been that at Mecca, Ohio. The wells are rarely over 100 feet deep; the oil has a density of 26° Baumé. Smith's Ferry, in Beaver County, Pennsylvania, has produced considerable oil of a peculiar amber color and about 28° Baumé. A number of shallow fields of West Virginia have been quite productive of heavy oils of excellent quality. One of the most noted was that at White Oak, West Virginia, where an excellent oil of 25° Baumé was produced at a depth of 160 feet. Nearly all the West Virginia oils are marketed without refining.

48. All heavy oils are by no means natural lubricants. Before being able to pass judgment on an oil, one must determine its purity, cold test, and fire test. The purity is estimated by mixing an average sample of the oil with an equal quantity of benzine, heating to about 120 F., and letting it settle for 6 hours in a graduated vessel. This gives a separation of all water and earthy matter. In Indiana, a heavy crude oil of 12° to 15° Baumé was produced containing large quantities of asphaltum, which rendered it practically valueless as a lubricant.

The variation in the cold test is quite marked in the heavy oils. The lower the cold test, the more valuable the oil. In this respect the oils from Mecca and Franklin are superior to all others.

A natural lubricating oil to be of real value must possess a fire test of at least 325° F. This is quite important, and many even of the low-gravity oils will not stand the requisite fire test for their employment in places where the inflammable vapors induced by friction would be dangerous. It has, therefore, been found necessary to bring these oils up to the standard by means of the processes called *sunning* and *reducing*.

49. Sunning. — In this process, heavy crude oils of 30° Baumé and under are selected. For this treatment, large, oblong tanks of about 18 inches in depth are employed. These are partially filled with water (about 6 inches) and the crude oil run in on this and exposed to the action of the sun and air for some time. This reduces the density and raises the fire test. It is one of the simplest processes to which natural oils are subjected.

50. Reducing.—The reducing process is similar to the foregoing. The large tanks are provided with steam coils, and not more than 4 or 5 inches of oil is placed on the water. The oil is heated to 110° F., when it becomes very limpid and every particle of impurity quickly subsides. The light oils evaporate and a crude of 32° Baumé is reduced to 29° Baumé in 2 or 3 days. The loss by evaporation depends

on the gravity and nature of the oil; if the crude contains much light oil, such processes will be too expensive, and therefore impracticable. In such cases it will be found better to place the oil in a still and carry out the process described under "Lubricating Still." Nearly all heavy cylinder oils are produced in this way.

It is found quite practicable to reduce oils of 30° and 32° Baumé in a still, since their value is enhanced to such an extent as to command an increase of several cents per gallon over the crude article. The proportion of natural lubricants, in comparison to those that have passed through a refining process, is very small. Under the latter head there are the steam-refined stocks, already described, and the treated and reduced distillates.

As previously mentioned, the manufacture of distilled lubricating oils and paraffin is really one industry. The wax oils and paraffin oils after pressing constitute the basis of all grades of treated machine oils. The wax oils are usually cut into approximately the oil that is wanted, when they are rerun to crystallize the paraffin. The common cuts are 38° to 34° Baumé, and 33° to 30° Baumé. The first cut is sometimes used without being pressed, as a filler in the manufacture of soap. The requirements for such an oil are freedom from acid and lye, 300° F. flash, and a pale straw color. When pressed, the oil is used as a basis for compounding miners' lamp oil. The State requirement for miners' oils is one of gravity; the requirement for this oil is a good color, cold test, and free-burning oil, and not lighter than 34° Baumé.

51. Neutral Oil.—The second cut of 33° Baumé is pressed and then reduced in a still, by means of fuel and steam, to about 32° Baumé and 400° F. fire test. The oil is again treated with acid and alkali in the lubricating agitator and filtered through bone-black filters. The finished product is known as *32° neutral oil.*

The first neutral oil was made by Merrill, of the Downer Oil Company. It received its name from being as nearly

neutral as it is possible to make a petroleum product. Merrill mixed 95 per cent. of his neutral oil with 5 per cent. of sperm oil, and thereby produced an oil in which the mixture could not be detected in color, odor, or taste. Oils of this type are used as mixing oils and for the lubrication of light machinery. Since the florescence of mineral oils is quite pronounced, it is common to expose these oils for some days in shallow tanks, in order to avoid their detection when used as mixing oils with vegetable or animal oils.

52. Paraffin Lubricating Oils.—The oil from the tar stills being run into one tank and pressed, is again rerun and cut into several fractions as paraffin lubricating oils. The common cuts are an oil of 29° to 30° Baumé, and a heavy cut of 23° to 26° Baumé. In order to give the oils a better fire test and viscosity, they are cut from ½° to 1° lighter than wanted and then reduced in the reducing still by means of steam and fuel. Thus the first cut is reduced to 28° Baumé of about 400° F. fire test, and the second made into 26°, 24°, and 23° Baumé oils, and 400° to 460° F. fire test. These paraffin oils are now pumped to a lubricating agitator.

53. Lubricating Agitator.—This agitator is similar to the one used for the treatment of illuminating oils, but is further provided with a steam coil for heating, and is usually surrounded with a brick jacket. Instead of carrying out the entire treatment in one agitator, it is customary to use two, one for the acid treatment and the other for the lye treatment. The cone is quite shallow and has large draw-off pipes to remove the acid sludge. The oil coming from the reducing stills is permitted to settle for some time to free it from sediment and moisture. The amount of acid used depends on the color desired, and the viscosity and gravity of the distillate; it varies from 4 to 15 per cent. of the oil treated. The average temperature for the treatment is 70° F., and it should not rise above 90° F.

The same rules hold good here that were to be observed in the illuminating-oil treatment; according as a high or

low temperature is used during treatment, so will the resulting oil be either dark or light in color. The method of treatment varies according to the preference and experience of the treater, some using all the acid in one charge and others dividing it into several portions. The following is a short description of a treatment in which the acid is divided into two portions.

54. Acid Treatment.—The oil is permitted to settle until it becomes transparent, and the water and sediment are drawn off; it is agitated and about one-third of the acid to be used added in the usual manner. The agitation is continued for about 1 hour. The sludge will soon gather in lumps, and after settling for 2 or 3 hours is drawn off as close as possible. It is wise to draw off this sludge as soon as possible, as it becomes absolutely solid after a short time.

The second acid is added in the same manner as the first and blown for about 1 hour. Some permit a settling of from 4 to 5 hours after the second acid has been blown, while others add 10 to 20 gallons of water to the 100 barrels of oil and thus throw the sludge acid out while the oil is still in agitation. The latter process is certainly much quicker; in either case the oil must be pumped from the acid agitator to the lye agitator. The sludge adheres to the sides of the agitator in such a manner as to make a good separation impracticable. A good wash cannot be carried out without great loss, and the addition of lye with the acid still remaining will bring the tarry matter into solution again.

55. Alkali Treatment.—The alkali agitator is constructed similarly to the acid agitator, and where practicable is placed at a lower level than the latter, in order to permit the oil to run from the former into it.

The neutralizing process with caustic soda is one of the most difficult processes connected with the treatment of lubricating oils, in which experience alone is a sure guide. The oil is agitated and 3 to 4 per cent. of caustic soda of 3° to 5° Baumé is added.

The oil loses its violet color and becomes a golden yellow as soon as it is neutralized. Samples taken out from time to time show signs of settling, and as soon as a good separation takes place readily, the agitation must be stopped.

The soaps are permitted to settle and, unless an emulsion has begun to form, can be drawn off in about 10 hours. During the agitation, one should test frequently for acidity and alkalinity. It is well to have the oil slightly alkaline, but still it must be remembered that a perfect neutrality is the point finally desired. When the settled soaps have been drawn off the oil is washed with hot water of 150° F. The aim is to leave it perfectly pure, and the washing must be continued until the water comes off perfectly clear and neutral. Great care and judgment must be exercised on the part of the treater to prevent loss. The oil is now run into large, flat settling tanks heated with steam coils, where it is kept until every particle of water has subsided and the oil has become perfectly clear and transparent. Agitation with warm, dry air assists greatly in this.

For light-colored and pale oils it is necessary to bleach them in shallow tanks or filter them through bone-black filters. The bleaching, or *debblooming*, as it is technically called, is carried out by similar methods to those already described under the heading "Chemical Treatment of the Distillate for Burning Oils." By this exposure its gravity is increased and its fire test lowered. Many so-called debloomed oils have not been subjected to this treatment, however, but have been debloomed by the addition of nitronaphthalene; such oils are severely condemned by engineers, as being ruinous to machinery. In order to still further improve the color of the oil, it may be passed through a bone-black filter in the filter house.

56. Filtering by Use of Animal Charcoal.—One of the modern appliances used in the production of high-grade lubricating oils is the animal charcoal filter. The filter house usually contains a number of filters arranged in benches, some of which are heated with steam and others by

means of natural gas or hot air. Tanks to be heated with steam are usually double-walled with a steam chamber between, while those to be heated with gas jets are suspended in a brick wall with a hollow space about the tank. In the latter case the entire space about the tank is heated, instead of applying heat directly to the tank. This avoids any overheating of the sides of the tank, which would injure the oil. By means of the hot-air system, flue gases may be utilized to good advantage.

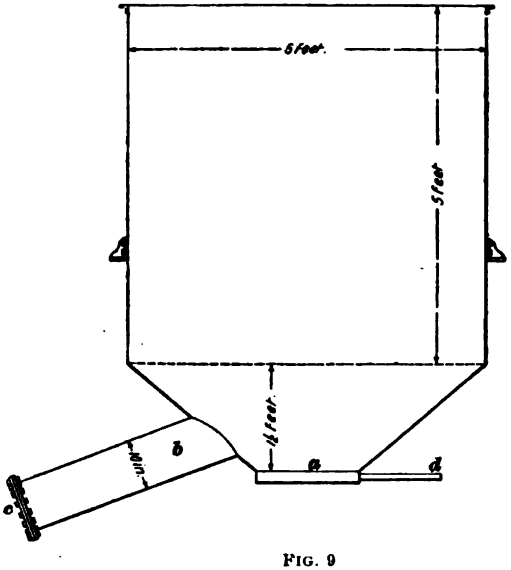


FIG. 9

57. Filters.—The tanks or filters are of two types, the side dump and bottom dump. Fig. 9 represents a side-dump tank. This is a well-riveted iron tank composed of $\frac{3}{16}$-inch iron. It is 5 feet in diameter and has a cone-shaped bottom with a perforated plate *a* about 4 inches above the outlet leading to the receiving tank. On the side, as represented, is a large pipe *b* reaching out from the tank, with a plate *c* bolted on to a smooth-fitting surface. By removing this plate the contents of the tank may be readily taken out.

The perforated bottom is covered with a thin layer of cotton batting and over this is about 2 feet of bone black. The oil is run in a slow stream on to the bone black, through which it filters and is then carried off through the 1-inch pipe *d*. It is provided with an automatic filler to prevent overflow in case the outlet of the filter should not act as fast as the feed. The oil is kept at about 120° F., which keeps the heavy oils perfectly limpid and aids the action of the bone black on the coloring matter contained in the oil.

The first oils coming off are always of a very light color and unless wanted of this shade for some special purpose it is mixed with the darker oils following, to some uniform color desired. As soon as the bone black ceases to accomplish the decolorizing wanted, a small car is run in alongside the filter, the plate on the side dump removed, and the bone black permitted to flow into the car, and the filter charged with a fresh lot.

58. Wash Filter.—The bone which has been removed is taken to the wash filter shown in Fig. 10. This, as is shown, is similar to the other filter except that it is stronger and provided with a screw-top man head *d*. The bone black is put into this and benzine forced in on it through the pipe *c* and permitted to filter through it and pass off to a tank or steam still through the 2-inch pipe *b*. This washing is continued until the benzine comes off perfectly water white. Steam is now forced into it through the pipe *a* and all benzine distilled off. The bone is now removed and put into cast-iron retorts and heated to dry and remove other impurities. This heating is very intense, but the admission of air is avoided.

This latter operation is so arranged that bone black is fed into the retorts and removed as a continuous process. In this manner the bone black is renewed, after which it acts as well as when used for the first time. Good bone black has thus been renewed as often as 116 times before it was found to be useless. When it is found to be impracticable

to attempt a further renewal of the bone black, it is sold to fertilizer factories. The benzine which has been used for the wash is distilled with steam and the residue used as a cold-test black oil. It is possible to obtain any shade of oil that is desired, from the perfectly white neutral oils to the finest grade of colorless vaselines and petroleum jellies.

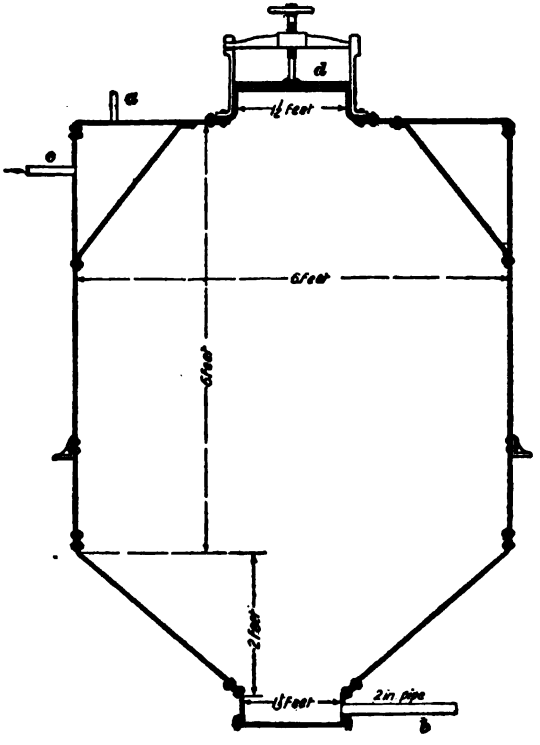


FIG. 10

59. In filtering the cylinder stocks, it is found that the first to come off have a very poor cold test and are of an almost white color. This is frequently used as a first-grade vaseline. Filtered cylinder stocks are always of a poorer cold test and viscosity than the unfiltered stocks from which they are made.

Many dealers prefer a filtered cylinder oil, but experienced engineers know that a dark, unfiltered cylinder oil is usually of much greater value as a lubricant than a light one. All that is necessary is to remove coke, suspended matter, and moisture, and all beyond that tends to reduce its value rather than improve it. The oil may gain in appearance at the expense of cold test and viscosity. This same rule will apply in the filtration of machine oils. Yet there are instances where a machinist may require an oil which is colorless and free from matter which would leave a stain.

Fullers' earth is being used very extensively in the filter house in place of bone black. Very frequently, illuminating oils are filtered through long filters of this earth, to change an off-colored prime-white oil into a water-white.

60. Low-Grade Lubricating Oils. — Besides these higher grades of lubricating oils there are a number of black oils manufactured for the lubrication of railroad cars and heavy machinery, where efficiency and cheapness are the only requirements. These black oils are usually produced in the same manner as the cylinder stock mentioned previously. The best crude oil is never used for this purpose, but now generally the Ohio and Indiana sulphur oils are used.

The common products are known as summer and winter black oils of 25°, 15°, and 0° cold test. The difference between the summer and winter black oils is simply a matter of congealing point. Since these oils are nearly all sold in tank-car quantities at the refinery, the purchaser places his special requirements on the oil desired; as a result, what is known as summer black to one purchaser may be a winter black to another.

The black car oil commonly used on freight cars is a black oil of 25° cold test and about 29° Baumé gravity. The oil known as 21° grease stock is made from Ohio crude and run with bottom steam to 21° Baumé gravity. It is used as a base for the manufacture of black axle and roll grease, and as a paving tar to be mixed with asphalt in street paving.

61. Table IV gives some of the more common pure mineral-oil lubricants manufactured in a refinery.

TABLE IV

Class of Oil	Degrees Baumé	Flash, Degrees F.	Fire, Degrees F.	Viscosity
32 neutral	32	365	415	*183
28 pale paraffin	28	370	425	*185
26 red	26	375	435	*195
23½ red	23.5	510	570	*300
Filtered cylinder	26	535	595	*135
600 cylinder	26 to 28	540	600	†171
650 cylinder	25 to 27	585	650	†190
700 cylinder	24 to 25	610	690	†215
Reduced black oils:				
Summer black	29	350	390	
Winter black	29	335	370	
25°–30° F. cold test	29	350	390	
15° F. cold test	30	325	360	
0° F. cold test	30	315	350	

* Tagliabue instrument. † Saybolt instrument.

62. Advantages in the Use of Mineral Oils as Lubricants.—Few users of lubricants fully appreciate the value of a mineral lubricating oil in comparison with the vegetable and animal oils formerly used exclusively. The advancement in the manufacture of petroleum and its lubricating products within the last decade have been especially marked. The cause of the slow gain in popularity was due to the opposition that the mineral oils met with when it was first attempted to gain recognition for them as lubricants. This brought about methods of testing the real economic value of oils and processes to improve deficient qualities and remove or greatly modify objectionable features. These tests have also drawn a marked line of distinction between the empirical manufacturer of machinery oils and the one who is

thoroughly familiar with the progress of scientific knowledge and the advances of manufacturing skill.

The modern lubricant manufacturer is desirous of knowing the particular purpose for which the oil is required, so that he can choose an oil, either pure mineral or compounded, which will give the best satisfaction for the purpose.

Mineral lubricants are used on all kinds of machinery; they are the safest and cheapest, and generally superior to animal and vegetable oils. They are safer on account of their high fire test, as they will resist great heat before vaporizing. They are more reliable, as they are usually pure, uniform in quality, and free from acid. The injurious effect of mineral oils on the metals of the bearings of any machine is less than one-half that of any pure vegetable or animal oil used as a lubricant.

Mineral lubricating oils are not affected by high-pressure steam or alkalies, while vegetable and animal oils are. The former last longer and work cleaner, they neither gum nor stain materials and manufacturers' products. It is not to be inferred from this, however, that animal or vegetable lubricating oils are driven from the market or are likely to be. They have distinct and important uses, and whether alone or combined with mineral oils, will continue to be employed for the purpose of lubrication.

63. Compounded Oils.—Although the pure animal and vegetable oils are falling somewhat into disuse, the compounded oils are becoming the principal lubricant for certain purposes; but it is impossible to lay down any fixed law that would enable an engineer to choose the right compound for any special machinery. The oils best suited for compounding with mineral oils are as follows:

Vegetable Oils	Animal Oils
Rape-seed	Neatsfoot
Olive	Lard
Palm	Tallow
	Sperm
	Whale

These oils when used in small quantities do not readily gum or become rancid. They usually increase the viscosity and staying quality of the oil and when not used in excess do not increase the action on the metal. Castor and cotton-seed oils are often used in compounds, but their use should not be encouraged, as they are far more liable to gum, and therefore retard rather than aid lubrication.

64. In the use of a lubricant, much depends on the intelligence of the engineer. He must know the speed, temperature, and work of his machine before he can form an idea of what oil would be best. Engineers have been known to work along using an oil either too light or too heavy for their work, without realizing that they could obtain an oil perfectly adequate in every respect for the same price or possibly less than that which they were paying. Cylinder oils are condemned as useless simply because engineers are using more oil than is necessary. Many engineers still hold that tallow is the only lubricant for steam cylinders and are constantly clamoring for a cylinder oil containing 25, 30, and even 50 per cent. of tallow oil.

Cylinder oils for low-pressure steam may be compounded with 5 to 10 per cent. of neatsfoot or tallow oil; but for high-pressure steam they should never contain more than 5 per cent. of animal oil. If the cylinder stock is strictly high grade, over 685° F. fire test, it has been found to give excellent satisfaction when entirely free from animal oil.

65. Effect of High-Pressure Steam on Animal Oils. Although the animal oil may lubricate a steam cylinder seemingly with general satisfaction, one must bear in mind that with high-pressure steam there is a decomposition, which is injurious to the cylinder and piston. The decomposition of tallow, for instance, is to form glycerine and stearic acid. Hot stearic, palmitic, or oleic acids readily attack copper and its alloys, as well as steel, iron, etc., and

therefore it is not difficult to see from the above how it is that steam cylinders and pistons are eaten away when a fatty oil, either by itself or in admixture with mineral oils, is used for lubricating them.

So great is the action, that there was removed from a 10″ × 12″ cylinder 2 pounds of a very hard, black substance that had been rolled into balls by the action of the piston. This substance contained 31 per cent. of fatty matter, 23 per cent. of moisture, and 46 per cent. of iron, and was the result of using a fatty oil lubricant. The piston and cylinder showed unmistakable proof of the action of the oil, although the latter had only been in use some 6 or 8 months, and the pressure of steam used in this case had not exceeded 40 pounds per square inch.

Instances similar to the above are of frequent occurrence about large manufacturing establishments, and a number of cases similar to this have come under the observation of the author where only a pure mineral oil would answer the purpose.

Heavy machine oils may be compounded with 10 per cent. of rape seed, olive, lard, or sperm oil.

Light machine oils are found to be better without any fatty oil.

Besides this class of compounded lubricating oils, there are oils compounded with soaps and solids for special purposes. In most cases they have been found very injurious to the machinery; while in a very few instances they are the most useful and valuable lubricants made today. The soaps commonly used are the aluminum and lead soaps. The addition of these soaps to a mineral oil increases the viscosity or body of the oil.

66. Mineral Castor Oil.—A very common oil made by the use of aluminum soap is a so-called *mineral castor oil.* It is an exceedingly viscous oil and may be used with a reasonable degree of satisfaction in some instances, yet in many places where it is used it would be better to use a pure mineral oil.

67. Galena Oils.—These oils, patented and first manufactured by Charles Miller, of Franklin, are compounded oils of a lead soap and Franklin heavy oil. It is probably the most noted compounded oil ever produced, its principal use being the lubrication of railway coaches.

68. *Graphite*, *soapstone*, and *mica* are used with mineral oils for special purposes. All these solids possess lubricating qualities within themselves, and when mixed with an oil become very efficient for the lubrication of excessively heavy and slow-running machinery.

69. Greases.—Greases having a semisolid to solid consistency are becoming a very important factor in the lubrication of certain kinds of machinery. Good greases are undoubtedly the best and most economical lubricants for machinery, if properly applied. The only drawback in their more general use is the difficulty experienced in applying them to certain bearings.

Greases are usually made from an insoluble soap mixed with a certain percentage of mineral oil. Insoluble soaps are those made from lime and rosin oil, cottonseed oil, or horse fat. They may be made solid or semisolid, according to the amount of mineral oil used in proportion to the soap. Under this head there are two classes of greases, *set* or *axle grease*, and *engine grease*. The former is made from a lime soap of rosin, mixed with a mineral oil. The latter is made from lime soap of horse fat or cottonseed oil, boiled with mineral oil. Both classes may be mixed with lead oxide, mica, or graphite.

70. Roll Grease.—Grease to be used on the rolls in the iron and tin-plate mills is frequently nothing other than wax tailings.

The list of compounded oils and greases is numberless; but their value is frequently not so much due to a particular percentage of composition as to the careful manipulation of the compounder.

THE MANUFACTURE OF VASELINE, WAXES, ETC.

71. Vaseline.—Vaseline was first introduced and patented by the Chesebrough Manufacturing Company, New York, and is, strictly speaking, a lubricating oil. It is made by the distillation of selected crude oil in the same manner as the cylinder stock already spoken of. The heavy cylinder stock, so to speak, is filtered through bone-black filters until the required color has been obtained; or the first filtration, which is of a light color, is used for vaseline, and the darker part used as filtered cylinder stock. The entire output of light cream-colored vaseline is bought up and controlled by the Chesebrough Manufacturing Company.

72. In order to bring the consistency and melting point to the proper degree, pure paraffin is melted and added to the filtered product. Its melting point is held at 86° F. or over. The United States Pharmacopœia gives the melting point at 104° to 125° F. Although the Chesebrough Manufacturing Company control the use of the name vaseline, other manufacturing concerns produce cosmolene, petrolene, and petroleum jellies, which differ only in name from vaseline.

The examination of Galician vaseline by Engler and Böhm is of considerable interest from a practical as well as from a scientific standpoint. The German Pharmacopœia considers a solution of paraffin in heavy oil as artificial vaseline. The examination of Engler and Böhm showed a marked difference between this imitation and the natural vaseline, a difference which is of interest to the commercial chemist, and one which can readily be made use of in the examination of a lubricating oil. The expansion on heating paraffin is much greater than that of vaseline. This difference in the coefficient of expansion is noticed in taking the gravity at different temperatures.

The natural vaseline shows very few crystalline bodies, where the artificial is quite crystalline; but on distillation the natural vaseline also becomes a mixture of crystalline paraffin and a heavy oil. The natural vaseline is thus shown to consist of a mixture of solid (not crystallizable) and liquid

hydrocarbons of almost identical percentage of composition and boiling points. This latter, in connection with the expansion coefficient and viscosity, are the distinguishing features which make the natural vaseline a more desirable article than the artificial. As a lubricant, the artificial is almost worthless.

73. Besides manufacturing vaseline from the best crude oil, much of it is made from the salvy residues of old tanks and pipe lines known as *B. S.* of the oil industry. The same product is obtained from the tubing and rods of pumping wells, and is known as *rod wax*. This refuse material is put into a still and heated with fuel and superheated steam, or, in some cases, air. In this manner the light oils still remaining are blown off. The remaining salvy mass is filtered through bone black, when it produces a vaseline of high melting point and good consistency. Thousands of barrels are thus produced each year from material which previous to this had been one of the greatest nuisances with which the producer had to contend.

The value of vaseline to the drug industry can hardly be overestimated. Vaseline, having no action or chemical affinity for most of the drugs, salves may be compounded with it which remain unchanged for years. Animal and vegetable fats and oils have been displaced almost entirely by its use. The "American Druggist" considers the following vaselines official:

Petrolatum Liquidum, liquid at ordinary temperature.

Petrolatum Molle, melting point 104° to 113° F.

Petrolatum Spissum, melting point 113° to 125° F.

Liquid petrolatum and albolene, made by McKesson and Robbins, are heavy oils practically devoid of paraffin, and carefully filtered to remove all color and odor. Any heavy machine oil with good cold test may be prepared in this way.

74. Waxes.—If the rod wax mentioned above be pressed directly instead of being put into the still and reduced, it will yield a low melting solid of a salvy half-paraffin nature.

This was the first material used in making chewing gum from any of the petroleum products. At present nearly all chewing gums are made by dissolving a certain percentage of either Chicle or Balata gum in a low melting and poorly crystalline paraffin as a basis, and to this is added some flavor or drug from which it receives its name and particular value.

Much of the very cheap gum is made without the addition of any gum. The petroleum product used is a compound, the nature of which lies between that of vaseline and paraffin.

Vaseline, amorphous, melting point 86° F.

Chewing-gum wax, partly crystalline, melting point 116° F.

Paraffin, crystalline, melting point 125° F.

PETROLEUM AND PRODUCTS

(PART 3)

TESTING OF PETROLEUM

1. Introductory.—These tests have reference to certain physical and chemical properties that the products of petroleum are required to possess in order to be considered of value in particular uses. In practice, as a rule, an examination of the physical properties is considered sufficient for the determination of the value of an oil; however, certain chemical properties are of no less importance in determining the real character of the oil. Most of these tests are carried out by means of especially constructed apparatus.

2. Specific Gravity.—The determination of the specific gravity is one of the tests that is considered important in every product from the lightest to the heaviest. It is considered important in the lighter products, since volatility is directly proportional to the degrees Baumé. In America the specific gravity is usually determined by means of hydrometers with the Baumé scale.

A weighing jar *A*, Fig. 1, is four-fifths filled with the oil to be examined; a hydrometer *B*, with a thermometer in the lower part, is introduced, and the depth to which the instrument sinks read off. In order to obtain accurate results, the instruments should be of the large type and graduated to $\frac{1}{10}$ degree. In practice, corrections for temperature are made by means of Tagliabue's "Manual for Inspectors of Coal Oil." These tables, however, are not perfectly reliable

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for light oils, and in cases where close results are required, it is best to weigh at 60° F. The specific gravity at 15.5° C. may be found by the formula

$$\text{Sp. Gr.} = \frac{140}{130 + \text{Baumé}}.$$

3. Testing of Gasoline or Benzine.—In the purchase or sale of gasoline and the light essences, the gravity is usually the only test that is made. It is a well-known fact to the refiner that the gravity requirement is of little consequence if he desires to produce a cheaper article. The free evaporating gasoline is what is wanted, and must be produced by avoiding all heavy naphtha. For instance, a stove gasoline of 70° Baumé, made from a cut between 74° and 66° Baumé, containing 9° in the cut, must be better than a gasoline made from lighter oil cut from 80° to 60° Baumé, containing 20° in the cut. A light crude will produce a better gasoline than a heavy oil, if the refiner will give the customer the benefit of a lighter product; but if he endeavors to increase his percentage and keep the gravity down, the consumer must suffer. The only test that will determine this evil is the distillation test, which is carried out in a similar manner to the distillation of the kerosene. Besides this, the gasoline must be odorless and free from acid. For boiling points, the student is referred to Table I.



FIG. 1

4. Odor and Acidity Test.—In order to test the odor of gasoline, take a piece of cloth, saturate it with the gasoline, and partially evaporate, when any foreign odor will become distinctive. The acid test is the same as that given under kerosene. Benzine to be used for pharmaceutical purposes, or for the mixing of paints and varnishes, is required to be odorless, free from acid and oil,

and from 60° to 63° Baumé. The test of the odor is taken similar to that of the gasoline.

5. Test of Benzine for Oil.—To test for oil, dip a sheet of writing paper into the benzine so as to cover about half the sheet, and permit it to evaporate. After evaporation, it should be perfectly free from any signs of grease and should have the same appearance when held towards the light as the part that was not saturated.

Another test is to burn a little of the benzine in a small evaporating dish and observe the amount of soot deposited on the sides of the dish and the color of the flame. If there is any soot after the burning, or a red-colored flame, it is a sure indication of oil in the benzine. No odor should be emitted while burning. The specific gravity is taken as usual.

6. Distillation Test for Benzine and Burning Oils. The same cautions regarding long cuts are true of benzine as well as gasoline, and the only means for determining this fact is by the distillation test. A good 63° Baumé deodorized benzine should distil between 248° and 302° F. The smallest fraction, if any, must pass over above 302° F. By referring to Table I, the approximate boiling points will be seen. The distillation test is the best test that can be applied to a burning oil in determining its value as an illuminant.

The amount of light oils is limited by the fire-test requirements, and the heavy oils, which are poorer illuminants in an ordinary lamp, must be determined by Engler's test, previously described. Instead of making a distillation of one fraction, as described in the test for the burning oil, it is well to divide the distillate into as small fractions as possible (every 50° F.). In a first-class illuminating oil, these fractions will be very nearly of equal volume.

It is not sufficient to know that an oil distils between 302° and 572° F., but also the percentage of the intermediate degrees should be known. For instance, an oil may be 48° Baumé, 150° F. fire test, and distil between 302° and

572° F. and yet be a very poor illuminant, on account of the cut at the still. It is possible to make an oil of a light fraction mixed with a heavy fraction that will have a good fire test and gravity and yet fail to make a free burning oil. An oil of that nature may burn well for a time and then refuse to burn on account of the heavy oil in its composition. The distillation will always reveal the manner in which the gravity and fire tests were made. The best kerosene is always made of the entire cut of the burning oil as it comes from the crude still. Much of the export oil is made from the cuts above and below the best water white. This is not only known to the American refiner, but has been distinctly shown by Engler in his distillation test.

7. The following are the results of tests on three different oils as given by Engler and C. Schestopal:

Alsace Brilliant petroleum. 801 Sp. Gr.
Kaiseroel (Imperial oil) 795 Sp. Gr.
Ordinary Pennsylvania petroleum. .800 Sp. Gr.

One hundred cubic centimeters of each were distilled, according to Engler's method. The results of these tests are given in Table I.

This does not mean that the Pennsylvania petroleum is inferior to the German oil, but that the American refiner has exported tops and bottoms and used the heart of his burning oil for some special oil to be consumed at home. The distillation is found to go beyond the limits, both above and below (302° to 572° F.). The light oil is not found to be injurious to the burning quality, but it injures the fire test. These distillation tests are coming into common use for the lighter as well as the heavier oils.

8. Flashing Point and Fire Test.—Petroleum consists of a mixture of hydrocarbons, and with a preponderance of light, volatile oils, its use is dangerous. When the readily volatile oils are not sufficiently separated by distillation, even at the ordinary temperature of a room, kerosene frequently

evolves vapors that form, with the air in the fount of the lamp, explosive gas mixtures. According to Chandler's experiments, the most violent explosion occurs with a mixture of 1 part petroleum vapor and 8 or 9 parts air; 1 part air and 3 parts petroleum vapor give a slight report, while a mixture of 1 part air and 1 part petroleum vapor does not explode.

TABLE I

Varieties of Oil	Boiling Point. Degrees F.	From Boiling Point to 302° F.	302°–347° F.	347°–392° F.	392°–437° F.	437°–482° F.	482°–527° F.	527°–572° F.	Above 527° F.	
Alsace Brilliant	296.6°	.8	10.5	20.00	24.70	20.00	15.4	4.8	3.80	cubic centimeters
			8.2	15.90	20.90	16.20	12.5	3.9		grams
Kaiseroel	212.0°		12.5	17.20	16.90	15.40	14.5	11.8	11.70	cubic centimeters
			9.6	13.30	13.50	12.50	12.0	9.6		grams
Pennsylvania kerosene	212.0°	15.8	10.0	12.00	8.25	11.00	7.8	9.0	26.15	cubic centimeters
		5.1	7.7	9.42	6.42	8.96	6.8	7.5		grams

To what degree a small percentage of naphtha will reduce the degree of temperature at which the oil evolves inflammable vapors is shown by the following experiments of Dr. C. B. White, of New Orleans, Louisiana. With an oil that originally yielded inflammable gases at 113° F., the respective degrees of temperature fell with the addition of 1 per cent. of naphtha to 103° F.; with the addition of 2 per cent. of naphtha, to 92° F.; with the addition of 5 per cent. of naphtha, to 83° F.; with the addition of 10 per cent., to 59° F.; with the addition of 20 per cent. of naphtha, to 40° F. The point at which these vapors will continue to burn when tested with a flame is called the *burning point*, or *fire test*. The *flashing point*, or point at which explosive vapors will rise, and the fire test can never coincide.

In testing illuminating oils, the flashing test is the most important, since it is the explosive vapors in small quantities that cause most of the accidents. Moreover, an oil with high flashing point is sure to have a high fire test, while the reverse is not true. Nearly all our States and foreign countries have regulations requiring a minimum flashing test, and in some cases both flash tests and fire tests.

9. Instruments for the Determination of Flashing Point.—A large number of instruments of various constructions are in use for testing the flashing point of oil. The results of a flash test vary according to certain conditions, viz., to the size of the oil surface and the distance between the flame or electric spark and the surface of the oil, and whether the oil is kept in motion or not. The two principal divisions under the testers are the closed and the open cups. The latter is the cup originally used; but for burning oils it has been almost superseded by the closed type.

The law was almost entirely put to naught on account of the ease with which the operator can influence his test. J. B. Marvin, M.D., says in his annual report, in regard to his experience with the open tester: "It was found that both the flash point and the burning point could be varied at the will of the operator, through a limit of several degrees, if the inspector is incompetent or careless."

10. Elliott Closed Cup. — One of the closed testers receiving considerable recognition by the various States is the Elliott cup, shown in Fig. 2, usually known as the *New York State Board of Health Tester*. It consists of a copper oil cup *d* holding about 10 ounces of oil—the quantity usually contained in lamps—heated in a water bath by a small alcohol lamp *a* or Bunsen flame. The cup is provided with a glass cover *c*, carrying a thermometer *b*, and a hole for the insertion of the testing flame, a small wax taper or wood splint, which gives a flame about the size of a pea. The New York State Board of Health gives the following directions for the use of the cup:

11. Determination of Flashing Point by Means of the Elliott Closed Cup.—"Remove the oil cup and fill the water bath with cold water up to the rivet mark on the inside. Replace the oil cup and pour in enough oil to fill it to within $\frac{1}{8}$ inch of the flange, or projection, joining the cup and the vapor chamber above. Care must be taken that the oil does not flow over the flange. Remove all air bubbles with a piece of blotting paper. Place the cork in the center hole of the glass top through which the thermometer passes, until its bulb is covered by the oil. Alcohol should be used in the lamp for heating the water bath, and the wick should be carefully trimmed and adjusted to a small flame. The rate of heating should be about 2° per minute, and in no case exceed 3°.

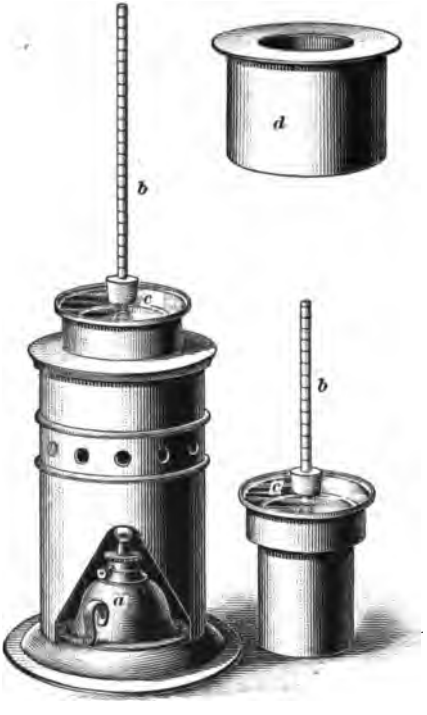


FIG. 2

"As a flash torch, a small gas jet $\frac{1}{4}$ inch in length should be employed. When gas is not at hand, employ a piece of waxed linen twine. The flame in this case, however, should be small. When the temperature of the oil has reached 85° F., the testings should commence. To this end insert the torch into the opening in the glass cover, passing it in at such an angle as to well clear the cover, and to a distance about half way between the oil and the cover. The motion should be steady and uniform, rapid, and without any pause. This should be repeated at every 2° rise of the thermometer

until the temperature has reached 95°, when the lamp should be removed and a test made for each degree of temperature until 100° is reached. After this, the lamp may be replaced, if necessary, and the testings continued for each 2°.

"The appearance of a slight bluish flame, as seen through the glass, shows that the flashing point has been reached. In every case note the temperature of the oil before introducing the torch. The flame of the torch must not come in contact with the oil. The water bath should be filled with cold water for each separate test and the oil from a previous test carefully wiped from the oil cup."

12. Tagliabue Closed-Cup Tester.—This instrument, also known as Tagliabue's coal-oil pyrometer, is a closed cup of practically the same design as the foregoing, only it is a much smaller cup, and on that account is considered inferior to some of the other types.

13. Foster Automatic Oil Tester.—This instrument is generally conceded by oil inspectors to be the only American instrument where the personal element is left out of question. It is strictly automatic and results must be concordant. The instrument is shown in Fig. 3, and consists of a copper lamp furnace *a* containing a water bath and oil cup. The latter is surmounted by a closed vapor chamber, which is pierced at two points symmetrically placed for the reception of a thermometer *b* and a flashing lamp, or taper *c*; the apparatus being elliptical in shape, the thermometer is placed in one focus of the ellipse and the flashing taper in the other.

The flashing taper consists of a small cylindrical wick holder, supported by radial arms to an annular ring, and rests upon a similar ring at the bottom of an open, shallow basin, the spaces between the radial arms giving egress to the oil vapor, while the wick itself extends down into the body of the oil within the cup. An inverted conical thimble, resting upon the rim of the basin, prevents the dissipation of the vapor. The thermometer is mounted in a copper tube, cut away in front to expose the scale; the bulb of the thermometer when in position is within the body of the oil

at a definite distance below the surface. An orifice around the tube of the thermometer, definite in diameter and distance above the surface of the oil, allows of a downward current of atmospheric air when the flashing taper is alight.

An index is placed within the water bath and the oil cup for maintaining uniformity in the filling of each. The heating lamp *d* of the lamp furnace has its wick adjustable, to facilitate uniformity in the rate of heating. All parts of the apparatus are definite and uniform in shape, size, and position. Each thermometer is of ascertained accuracy, and if instructions for using are carefully followed, the most accurate results are attainable.



FIG. 3

14. Determination of Flashing Point by Means of the Foster Automatic Tester.—In determining the flashing point by means of the Foster automatic tester, proceed as follows:

1. Remove the thermometer with its mounting from the oil cup.

2. Lift off the oil cup containing the flashing taper and fill the open water bath with water, to the mark upon the inside.

3. Now take out the wick holder from the oil cup and fill this vessel with the oil (at 60° F.) to be tested, pouring in the oil at the place of the wick holder and noting the gauge mark at the thermometer hole; pour in the oil very gradually as soon as the surface of it approaches the gauge mark. The gauge mark consists of a small pendent shelf, and the oil cup is properly filled when the upper surface of the oil just adheres to the lower surface of the gauge mark.

Too much care cannot be taken at this point; therefore, after having ceased pouring, tip the cup so that the oil flows away from the gauge, and then, gradually restoring it to the horizontal, see that the surface again adheres and add a little more oil if it does not.

4. See that the wick of the flashing taper be adjusted to give a very small flame—a flame that does not exceed ¼ inch in height. A flame that exhibits as much blue at its base as yellow at its top is proper.

5. Now set the oil cup on top and into the water bath; return the flashing taper to its place, inverting the conical thimble around it, and return the thermometer to its place upon the cup; in doing this, be sure that the casing of the latter is pushed down upon the cup as far as it will go.

6. Fill the lamp beneath half full of alcohol, light it, and put it in its place beneath the water bath. Now note the rate of increase in temperature as shown by the thermometer, and adjust the wick to raise the temperature at the rate of 2° per minute. When the temperature has reached 100°, light the flashing taper and observe it closely. As soon as the oil under test has reached its flashing point, the flame of this taper will be extinguished by the first flash, and the point of attention is to note the temperature at the instant the flame of the taper is extinguished. This *flashing point* is the point of temperature at which the oil generates a vapor, the composition and character of which is that of ordinary illuminating gas. The *flash* indicates that this has formed an explosive mixture with atmospheric air.

15. Determination of Fire Test by Means of the Foster Automatic Tester.—The fire test is made by continuing to heat the oil (the cover being removed in the case of a closed tester) at the same rate after the flash test is made and flashing the oil every 4° until the flame remains. The fire test of illuminating oils is usually from 15° to 25° F. higher than the flash test of the open cup.

In testing 300° oil—*Mineral Seal* or *Miners' Sperm*—the test is usually made in an open cup of brass. The oil is

heated at the rate of 5° per minute and the test flame applied every 5° after 230° F. has been reached. The flash point should not be under 250° F. in the open-cup test. By comparing these cups and looking over the requirements of the different States, it will be seen that the refiners' sales of illuminating oils are of great variety. Each refiner is compelled to have on hand a number of reliable testers, but as these cups are not used regularly by the official oil inspector, the latter's tests in many cases will be unreliable. Every refiner has striven to reduce his tests to one instrument, using the one in which the error from the operator's use is reduced to a minimum. Tables II, III, and IV are attempts to give some comparison of the different testers.

16. Abel Closed-Cup Oil Tester.—The apparatus shown in Fig. 4 (*a*) and (*b*), adopted by England, Germany, and Austria, is strongly recommended, as it is of importance to the American refiner and exporter, all export oil being tested by it. The instrument and its uses are thus described in the Schedule of the 1879 Petroleum Act of the British Parliament.

The oil cup *a*, Fig. 4 (*b*), consists of a cylindrical vessel 2 inches in diameter, $2\frac{3}{10}$ inches high (internally), with outward projecting rim *b*, $\frac{5}{10}$ inch wide, $\frac{3}{8}$ inch from the top, and $1\frac{7}{8}$ inches from the bottom of the cup. It is made of gun metal or brass. A bracket *c*, consisting of a short, stout piece of wire bent upwards and terminating in a point, is fixed to the inside of the cup, to serve as a gauge. The distance of the point from the bottom of the cup is $1\frac{1}{2}$ inches. The cup is provided with a close-fitting, overlapping cover *d* made of brass, which carries the thermometer *e* and test lamp *f*. The latter is suspended by two supports from the side by means of trunnions, upon which it may be made to oscillate; it is provided with a spout, or wick tube, the mouth of which is $\frac{1}{16}$ inch in diameter. The socket to hold the thermometer is fixed at such an angle and its length is so adjusted that the bulb of the thermometer, when inserted to full depth, shall be $1\frac{1}{2}$ inches below the center of the lid.

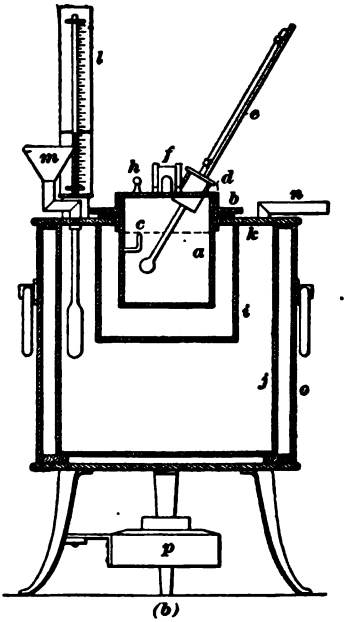
The cover is provided with three rectangular holes: one

in the center, $\frac{5}{10}$ inch by $\frac{4}{10}$ inch, and two smaller ones (not shown in the figure), $\frac{3}{10}$ inch by $\frac{2}{10}$ inch, close to the sides and opposite each other. These three holes may be closed and uncovered by means of a slide *g* moving in grooves, and having perforations corresponding to those on the lid. In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and



(a)

FIG. 4



(b)

FIG. 4

tilted in such an angle as to bring the end of the spout just below the surface of the lid. Upon the slide being pushed back so as to cover the holes, the lamp returns to its original position. Upon the cover, in front of, and in line with, the mouth of the lamp, is fixed a white bead *h*, the dimensions of which represent the size of the test flame to be used. The bath, or heated vessel, consists of two flat-bottomed copper cylinders, an inner one *i*, 3 inches in diameter and

2½ inches in height, and an outer one *j*, 5½ inches in diameter and 5¾ inches in height. They are soldered to a circular copper plate *k*, perforated in the center, which forms the top of the bath, in such a manner as to enclose the space between the two cylinders, but leaving access to the inner cylinder. The top of the bath projects both outwards and inwards about ⅛ inch, that is, its diameter is about ¼ inch greater than that of the body of the bath, while the diameter of the circular opening in the center is about the same amount less than that of the inner copper cylinder. To the inner projection of the top is fastened, by six small screws, a flat ring of ebonite, to avoid metallic contact between the bath and the oil cup. The exact distance between the sides and the bottom of the bath and of the oil cup is ½ inch. A split socket, similar to that on the cover of the oil cup, but set at a right angle, allows a thermometer *l* to be inserted into the space between the two cylinders. The bath is further provided with a funnel *m*, an overflow pipe *n*, and two loop handles. The bath rests upon a cast-iron tripod stand, to the ring of which is attached a copper cylinder, or jacket *o*, flanged at the top and of such dimensions that the bath, while firmly resting on the iron ring, just touches with its projecting top the inward turned flange. The diameter of this outer jacket is 6½ inches. One of the three legs of the stand serves as a support for the spirit lamp *p* attached to it by means of a small swing bracket. The distance of the wick holder from the bottom of the bath is 1 inch.

Two thermometers are provided with the apparatus, one *l* for ascertaining the temperature of the bath, and the other *e* for determining the flashing point. The thermometer *l* for ascertaining the temperature of the water has a long bulb and a space at the top. Its range is from about 90° to 190° F. The thermometer is adjusted with a metal collar fitting the socket, and the part of the tube below the scale should have a length of about 3½ inches, measured from the lower end of the scale to the bulb. The thermometer *e* for ascertaining the temperature of the oil is fitted with collar and ivory scale in a similar manner to the one described. It has a

round bulb, a space at the top, and ranges from about 55° to 150° F.; it measures from end of ivory back to bulb 2¼ inches.

17. Determination of Flashing Point by Means of the Abel Tester.—The test apparatus should not be placed for use in a position where it is exposed to currents of air, or drafts. The heating vessel, or water bath, is filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the commencement of the test is to be 130° F. When a test has been completed, this water bath is again raised to 130° by placing the lamp underneath, which is done while the petroleum cup is being emptied, cooled, and refilled with a fresh sample to be tested. The lamp is then turned on its swivel from under the apparatus and the next test is proceeded with.

The test lamp is prepared for use by fitting it with a flat-plaited candle wick and filling it with colza or rape oil up to the lower edge of the opening of the spout or wick tube. The lamp is so trimmed that when lighted it gives a flame about .15 inch in diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil cup, is readily maintained by simple manipulation from time to time with a small wire trimmer. When gas is available, it may be conveniently used in place of the little oil lamp, and for this purpose a little test-flame arrangement for use with gas may be substituted for the lamp.

The bath having been raised to the proper temperature, the oil to be tested is poured into the petroleum very slowly until the level of the liquid just reaches the point of the gauge fixed in the cup. In warm weather, the temperature of the room in which the samples to be tested are kept should be observed in the first instance, and if it exceeds 65° F., the samples to be tested should be cooled down (to about 60°) by immersing the bottles containing them in ice water. The lid of the cup, with the slide closed, is then put on and the cup is placed into the bath, or heating vessel.

The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid and must not be altered. When the cup has been placed in the proper position, the scale of the thermometer faces the operator.

The test lamp is then placed in position upon the lid of the cup, the lead line, or pendulum (the pendulum should be 24 inches in length from the point of suspension to the center of gravity of the weight), which has been fixed in a convenient position in front of the operator, is set in motion and the rise of the thermometer in the petroleum cup is watched. When the temperature has reached about 66° F., the operation of testing is to be commenced, the test flame being applied once for every rise of 1° in the following manner: The slide is slowly drawn open while the pendulum performs three oscillations, and is closed during the fourth oscillation.

NOTE.—If it is desired to employ the test apparatus to determine the flashing points of oils of very low volatility, the mode of proceeding is to be modified as follows: The air chamber that surrounds the cup is filled with cold water to a depth of 1½ inches, and the heating vessel, or water bath, is filled as usual, but also with cold water. The lamp is then placed under the apparatus and kept there during the entire operation. If a heavy oil is being dealt with, the operation may be commenced with water previously heated to 120° F., instead of with cold water.

18. Tagliabue Open-Cup Tester. — Under the open testers, Tagliabue's open cup, shown in Fig. 5, is the most important. It was the official testing apparatus in England and many of the United States, but, as has been previously stated, has been superseded almost entirely by the closed type. It is similar in construction to the closed cups, having a brass water bath *a* and a glass oil cup *b*, but no cover.

19. Determination of Flashing Point by Means of Tagliabue's Open-Cup Tester.—The water bath is nearly filled with cold water, allowing for the displacement by the oil cup; the latter is filled to within $\frac{3}{32}$ inch of the top, care being taken not to wet the rim, and the thermometer placed in position. The heating flame is adjusted so that it is ¼ inch high, and the heating proceeded with at the rate of

2½° per minute, until 97° F. is reached, when the test flame is applied and testings made every 3° until the flash point is reached. The test flame should be that of a waxed linen twine, wood splint, or gas jet. It is slowly and carefully passed over the kerosene about ½ inch from the surface. The test depends very much on the position and size of the flame.



FIG. 5

Tables II, III, and IV show a comparison of the different results obtained with different testers.

20. Color Test for Illuminating Oils. The color test of illuminating oil is usually carried out in a 4-ounce sample bottle in which the oil is compared with standard colors prepared by means of solutions of potassium chromate in water. In America, the colors in use are: (1) Water white, (2) prime white, (3) standard white. In England (i. e., receiving ports): (1) Water white, (2) prime white, (3) standard white, (4) merchantable. In Germany (i. e., receiving ports): (1) Water white, (2) prime white, (3) standard white, (4) prime light straw to white, (5) prime light straw to standard, (6) light straw, (7) straw. The colors in the foreign countries are usually determined by some colorimetric apparatus.

21. Cloud Test. — This test is made as follows: Two ounces of the oil are placed in a 4-ounce sample bottle, with a thermometer suspended in the oil. The bottle is exposed to a freezing mixture of ice and salt, and the oil stirred with the thermometer while cooling. The temperature at which the cloud forms, due to crystallization of paraffin, is taken as the cloud test. Aside from the most common tests previously mentioned, it is very often necessary to make certain tests in regard to the purity of the oils.

22. Preliminary Acid Test.—Shake equal quantities of hot water and oil in a test bottle, pour off the oil, and test the water with litmus solution. The acid in this case is usually sulphuric, coming from the imperfect refining.

TABLE II

COMPARISON OF COMMON TESTERS

Foster Closed-Cup Flash. Degrees F.	Tagliabue's Closed-Cup Flash. Degrees F.	Elliott Closed-Cup Flash. Degrees F.	Tagliabue's Open-Cup Flash. Degrees F.	Tagliabue's Open-Cup Fire. Degrees F.	Grade of Oil
85	72	67	90	100	
90	77	70	100	110	110° oil
94	82	76	110	117	120° oil
100	89	82	109	125	
106	97	87	112	130	130° oil
111	100	94	115	135	135° oil
115	107	100	120	140	140° oil
120	115	108	124	147	150° oil
133	129	117	135	159	160° oil
146	142	130	154	170	175° oil

23. Qualitative Test for Sulphur.—In testing for sulphur in the imperfectly refined oils of Ohio, it is customary to treat a small sample with sodium plumbate. Any change of color indicates sulphur. Other compounds of sulphur may be detected by heating the oil to its boiling point with a bright piece of sodium or potassium. If sulphur compounds be present, a yellowish layer is formed upon the metal. After cooling, add distilled water, drop by drop, until the metal is dissolved, and test for sulphides with sodium nitroprusside. A fine violet color indicates sulphur. Aufrecht makes the following statement: "Good petroleum should not contain more than 10 to 15 milligrams of sulphur in 100 cubic centimeters."

24. Quantitative Estimation of Sulphur in Petroleum.—To 50 cubic centimeters of the petroleum, .5 gram

TABLE III

COMPARISON OF TESTERS (ENGLER AND HASS)

Name of Tester	Sample A				Sample B				Sample C			
	Number of Tests	Results. °C.	Mean. °C.	Difference. °C.	Number of Tests	Results. °C.	Mean. °C.	Difference. °C.	Number of Tests	Results. °C.	Mean. °C.	Difference. °C.
Tagliabue open..	1	22.7 to 38.8	30.95	16.1	9	32.2 to 48.8	42.00	16.6	6	45.5 to 57.2	52.20	13.3
Tagliabue closed.					18	24.0 to 39.4	31.68	15.4				
Danish.....	5	19.5 to 21	20.80	3.5	4	29 to 31	30.00	2.0	4	42 to 45	43.25	3.0
Saybolt.....	4	36.6 to 36.7	36.30	1.1	2	36.1 to 36.6	36.35	.5	2	48.5 to 52.7	50.75	3.9
Parrish.....	5	20.7 to 23	21.40	2.7	15	25.5 to 30.7	27.30	5.2	9	36.5 to 39	37.70	2.5
Abel.....	4	16 to 17.1	16.60	1.1	7	22.2 to 23.8	22.64	1.6	3	32.4 to 33.8	32.96	1.8
Engler.....	4	21 to 22.5	21.95	1.5	19	28 to 30.5	29.40	2.5	2	39.3 to 39.7	39.50	.4

TABLE IV

(REDWOOD)

Oil Sample	Tagliabue Open Cup. Degrees F.	Arnabaldi Open Cup. Degrees F.	Saybolt. Degrees F.	Tagliabue Closed Cup. Degrees F.	Tagliabue Closed Cup. Large Size. Degrees F.	Wisconsin. Degrees F.	Abel Closed Cup. Degrees F.	Henstein. Degrees F.	Millap. Degrees F.	Mann. Degrees F.	Foster Automatic Closed Cup. Degrees F.
1	110	118	120	111	117	107	103	130	111	95	119
2	111	121	124	115	118	107	102	128	107	96	
3	119	122	122	112	118	108	102	130	108	95	118
4	97	96	97	90	93	86	76	90	81	75	96

of sodium bicarbonate is added, and the mixture is distilled at the rate of 40 to 50 drops a minute until about 45 cubic centimeters is distilled over. The residue is placed in a large porcelain dish and repeatedly washed with ether; after spontaneous evaporation of the latter, about .5 gram of sodium is added in small pieces and the whole evaporated over a small flame to a sirup, and then ignited with the gradual addition of ammonium nitrate until the ash is quite white. The residue is extracted by very dilute hydrochloric acid and the sulphuric acid estimated in the filtrate in the usual manner by barium chloride.

25. Sulphuric-Acid Refining Test. — The object of this test is to judge the degree of refinement to which the oil has been subjected, a well-refined oil giving little or no color when submitted to this process. A glass-stoppered test bottle is filled half full of the oil and about half as much chemically pure sulphuric acid of 66° Baumé

added. This mixture is then shaken for several minutes. The depth of color indicates the impurities still remaining in the oil. In comparative tests this color is matched by various standard solutions of Bismarck brown.

26. Alkali Refining Test.—In order to reduce expenses, attempts are frequently made to employ less alkali than really is required. Although the oil shows a good color for a short time, this disappears after a little time and its illuminating properties are impaired. The test is made similar to that of the sulphuric-acid test, using a caustic-soda solution of about 22° Baumé and observing the change of color.

THE TESTING OF LUBRICATING OILS

27. Cleveland Open-Cup Fire Tester. — In testing lubricating oils, the open cup is used almost exclusively. The one shown in Fig. 6 is known as the *Cleveland open-cup fire tester*, and is commonly designated by purchasers of lubricants as the one to be used in making the flash tests and fire tests of the oils. It is made of heavy brass throughout with a solid glass thermometer. The cup *a* is filled with oil to within $\frac{5}{8}$ inch of the flange, and in case of cylinder oils, to $\frac{1}{2}$ inch. The flange is carefully dried with blotting paper. The thermometer *b* is so placed as to have the bulb completely covered with oil. The gas flame is regulated so as to heat the oil at the rate of 15° F. per minute. The testing flame should be first applied at 50° F. below the supposed flash test, and then every half minute until the flash point is reached. In making the fire test, heating is continued and the flash test made every half minute until it remains burning.

28. Specific Gravity.—The specific gravity of cylinder oils is taken in a manner similar to that of illuminating oils, but must be at a higher temperature and the reduction

made according to Tagliabue's "Manual for Inspectors of Coal Oil."

29. Viscosity Tests.—The viscosity of a lubricant is usually considered its most valuable quality. This is usually taken by means of the apparatus called a *viscosimeter*

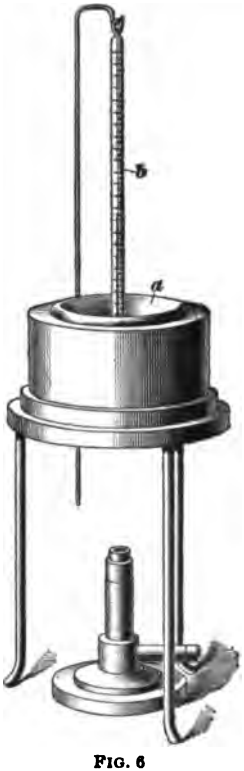


FIG. 6



FIG. 7

(see *Quantitative Analysis*). A great variety of these have been constructed, all based upon the length of time required for a given quantity of oil to flow through a small orifice, as compared with water or some other oil used as a basis. The best-known instruments are those of Saybolt, Tagliabue, and Engler.

30. Saybolt Viscosimeter.—This instrument, which is under the control of the Standard Oil Company, is made in three forms—A, B, and C. Apparatus A is the standard for testing distillates at 70° F.; B, for testing the light, black oils at 70° F.; and C, for testing heavy, black oils and cylinder oils at 212° F.

31. Tagliabue Improved Viscosimeter.—The Tagliabue improved viscosimeter, shown in Fig. 7, is generally used by the independent refiners. The instrument as shown consists of two metallic tanks, or receptacles *A* and *B*. The lower one *A* is a water boiler, while *B* is a vessel in which is placed the receptacle containing the oil to be tested. Direct communication is established between *A* and *B* by means of the stop-cock *b*. The stop-cocks *d* and *e* and the coupling *g* communicate with the interior of *B*. The stop-cock *h* communicates with the oil receptacle, so that the oil may flow out through nipples of specified sizes attached to it. The thermometer is suspended in the oil as shown. When testing at 212° F., water in *A* is boiled and steam passes through the stop-cock *b* into *B*, thus warming the oil in the receptacle to the required temperature. When testing at lower temperatures, *b* is closed and water heated to the required temperature is poured into *B*.

32. Test for the Viscosity of Oils at 212° F.—Pour water into boiler *A* through opening *a*, after unscrewing the safety valve, until the water gauge shows that the boiler is full. See that stop-cock *b* is open, making direct connection between the boiler and the upper vessel that surrounds the receptacle in which the oil to be tested is placed. Place a wire holder in the set nut *c* and suspend a thermometer so that its bulb will be about $\frac{1}{4}$ inch from the bottom of the oil bath. Then, after carefully straining 70 cubic centimeters of the oil to be tested, which, of course, must be warmed in the case of very heavy oils, pour the same into the oil bath. Close stop-cocks *d* and *e*. Screw the extension *f*, with rubber hose attached, into coupling *g* and let the open end of the

hose be immersed in a vessel of water, which will prevent too large a loss of steam. Place a lamp or a Bunsen burner under the boiler, screw the steel nipple, furnished with the apparatus and marked 212, on to stop-cock *h*, and the apparatus is ready to use.

After steam is generated, wait until the thermometer in the oil bath shows a temperature of from 209° to 211° F.; then place the 50-cubic-centimeter test glass under stop-cock *h* so that the stream of oil strikes the side of test glass, thereby preventing the formation of air bubbles, and, when the thermometer indicates its highest point, open the cock *h* simultaneously with the starting of the watch, which is supplied with each instrument. When the running oil reaches the 50-cubic-centimeter mark in the neck of the test glass, the watch is instantly stopped and the number of seconds noted. Then multiply the number of seconds by two, and the result will be the viscosity of the oil. For example, if 50 cubic centimeters of oil runs through in 101½ seconds, the viscosity will be 203. It is necessary to keep the oil well stirred before making the test, in order to have the oil at a uniform temperature.

33. Test for the Viscosity of Oils at 70° F.—Screw steel nipple, furnished with the apparatus and marked 70, on to faucet *h*, and close stop-cock *b*, closing communication between the boiler and the upper vessel; also close stop-cock *e*. Fill the upper vessel through opening *g* with water at a temperature as near 70° F. as possible; also have the oil to be tested at same temperature. Hang the thermometer in position, and after stirring the oil thoroughly, blow through rubber tube at *d*, to thoroughly mix the water. Should the thermometer show higher or lower than 70°, add cool or warm water until the desired temperature is attained. Then proceed as before.

NOTE.—In making tests at 70° F., it is absolutely necessary that the temperature of the oil should be fully 70° before starting the run.

34. Engler Viscosimeter.—This instrument is commonly used in Europe, and although the viscosity numbers

differ from the foregoing, it is constructed on a similar principle.

35. Cold Test.—The cold test of an oil is that point at which it will not flow freely from a vessel. In order to make this test, put about 1 ounce of the liquid to be tested into a common 4-ounce sample bottle and place a short, stout thermometer in it. Then place the bottle in a refrigerator, where the liquid will become frozen. When the liquid has become solid throughout, remove from the refrigerator and allow the liquid to soften, thoroughly stirring and mixing it at the same time, by means of the thermometer, until the mass will run from one end of the bottle to the other. Now grasp the bottle by the neck, having in the same hand a little waste or a towel, which encloses the thermometer, withdraw the thermometer through the waste, to wipe it far enough to see the mercury, and read the temperature. The reading is the cold test of the liquid.

36. Chilling Point.—The chilling-point test is employed where it is desired to know whether the liquid remains clear at any given temperature. Use a similar bottle and thermometer and the same amount of liquid as for cold test. Expose the liquid to the given temperature, or preferably a little below; stir it with the thermometer occasionally, until the temperature of the whole liquid becomes the one desired, and then allow it to stand for the time specified; at the end of which observe whether the liquid remains transparent and free from flocks and scales of congealed constituents. If so, it stands the test, or chilling point.

37. If it becomes necessary to know at what temperature flocks, or scales, of congealed constituents, or more or less opacity actually takes place, proceed as follows: Use the same kind of bottle and thermometer and the same amount of oil as for the cold test. Expose the oil to a temperature 5° colder than that at which the liquid remained perfectly clear, that is, free from flocks and scales, and allow

the temperature to fall below that, with constant watching and occasional stirring. Lower the source of cold 5° more and treat as before. Continue until opacity, or flocks or scales, begin to show. The reading of the thermometer when this point is reached is the temperature sought. It is well to determine a few of the intermediate points of solidification, such as the temperature, when it just begins to solidify, the temperature at which it will stick to the thermometer, like vaseline, and the temperature when it becomes hard, like butter.

38. Congealing Point.—For soap stock, the following test has been used: A 4-ounce sample bottle half filled with a sample of oil, with a thermometer inserted, is placed in a freezing mixture and chilled. The point at which the thermometer becomes stationary for some time before the mass becomes solid is considered the *congealing point.* This point is always higher than the cold test.

39. Freezing Mixtures.—Quite a number of methods have been proposed for obtaining the low temperature to be used in taking cold tests and chilling points. However, none of these methods is apparently so simple, so easily manipulated, and so expeditious as freezing mixtures. Three cases are to be considered:

1. *Mixtures for Cold Tests.*—If the cold test is above 35° F., use a mixture of snow or pounded ice and water in any suitable non-conducting vessel. A good-sized vessel and a plentiful supply of snow or pounded ice works best. The bottle with oil and thermometer is plunged into the freezing mixture and allowed to remain there until the liquid is frozen solid.

For cold tests from 35° F. down to zero, use a mixture of 2 parts, by weight, of snow or pounded ice and 1 part of common salt. The same remarks as above in regard to vessel, plentiful supply, and manipulation apply.

For cold tests from 0° F. to — 30°, use a mixture of 3 parts, by weight, of crystallized calcium chloride and 2 parts of

snow or very finely pounded ice. The crystallized calcium chloride, if obtained in the market, should be crushed in a mortar into pieces not larger than wheat kernels. For cold tests approaching the lower range of the above limit, the vessels should be non-conducting and should be cooled to 32° F. before putting in the calcium chloride and ice. The crystallized calcium chloride may be bought in the market, but the following method gives a very cheap and efficient article from materials always at hand in every good laboratory, viz.:

Put about 1 pound of pulverized carbonate of lime (marble dust works best) into a casserole holding from 3 pints to 2 quarts, and pour concentrated commercial hydrochloric acid on it, a little at a time. A neutral solution of calcium chloride is formed very quickly, after each addition of acid, and in a short time the mass becomes wet enough to be stirred, to facilitate the action. Proceed with the addition of the acid until nearly all the marble dust is dissolved, taking care to avoid an excess of acid. Pouring the acid on to the marble dust, instead of adding the dust to the acid, prevents the suffocating fumes of HCl that are sure to be given off if the latter procedure is followed.

After the last addition of acid, filter into a convenient metal vessel whose weight is known; boil until a drop of the liquid placed on a watch glass and cooled by placing the glass on a lump of ice becomes solid rather quickly; then cool by placing the vessel in another vessel containing ice and water. As the temperature falls the calcium chloride should be stirred, to prevent the formation of a compact mass that is not easily broken up. If the operation is properly performed, when the temperature of the calcium chloride reaches 40° F. or a little below, the material in the metal vessel will be a more or less mushy mass of calcium-chloride crystals.

Now remove the metal vessel from the ice water, wipe it dry on the outside, and weigh, to determine the amount of calcium chloride. Wrap the bottom and sides of this vessel

well with towels or other non-conducting material, and then for every 3 parts of calcium chloride add 2 parts of snow or finely pounded ice and stir thoroughly. After a minute or two, the material to be tested can be placed in the liquid, and the whole thing should then be covered, to prevent access of heat.

2. *Mixture for Cloud Tests.*—When it is desired to find out whether a liquid remains clear at any given temperature, the following is recommended:

(*a*) For temperatures above 32° F., place 1 gallon of water in a wooden bucket and regulate the temperature by adding either ice or cold water. Care should be taken to keep the water at the desired temperature while the liquid in the sample bottle is cooling.

(*b*) For temperatures from 32° F. down to zero, add to 1 gallon of water, in a bucket of wood or indurated fiber, 15 pounds of cracked ice. Stir thoroughly with a wooden stick, and when the temperature has reached 32° F. or thereabouts, add, with continued stirring, sufficient dry common salt to produce the temperature desired. Approximately, each quarter pound of salt added will lower the temperature under the conditions given 2° F., until 10° is reached, when twice the quantity must be added to bring the temperature down 2°.

As the usual specified temperatures at which chilling points are observed are 32°, 20°, 10°, and 0° F., it is fairly safe to say that these temperatures may be obtained as follows: For 32° F., into a wooden or indurated-fiber pail put 1 gallon of water and any convenient amount of ice, provided only sufficient is used; for 20° F., 1 gallon of water, 15 pounds of cracked ice, and 1½ pounds of dry common salt; for 0° F., ½ gallon of water, 15 pounds of cracked ice, and 5 pounds of dry common salt.

A little experience will enable the desired temperatures to be reasonably well controlled. Of course, the melting of the ice continually dilutes the salt solution, and if a fairly

constant temperature for some time is desired, occasional small additions of salt will be necessary. For temperatures below 0° F., crystallized calcium chloride may be used in place of common salt. So much depends on the amounts of the materials used (especially when dealing with such low temperatures), on the appliances, and the protection given to the vessels in which the cold is produced that it is perhaps hardly wise to try to give proportions. It may be said, however, that 3 pounds of finely crushed ice and 2 pounds of crystallized calcium chloride in a properly cooled and protected vessel will give 10° below zero F., and 3 pounds of finely crushed ice and 3¾ pounds of crystallized calcium chloride under the same conditions will give 20° below zero F. A few experiments, however, are worth more than a good many directions.

3. *Mixtures for Chilling-Point Tests.*—When it is desired to know at what temperature flocks, or scales, of congealed constituents of more or less opacity take place, use the methods of obtaining desired temperatures described in the preceding paragraph, except that in going down the scale, temperatures 5° apart are produced by the proper mixtures and used as described.

40. Evaporation Test.—The object of this test is to determine what percentage of the oil is volatile when exposed to the conditions existing on the bearings to which the oil is to be applied. It may be carried out by means of a small quantity of oil on a watch glass, the whole first being accurately weighed. The watch glass with oil is placed in an air bath and subjected to the temperature required (light oils should stand a test of 150° F. for 8 hours), and cooled and reweighed. The loss being figured in per cent. represents the amount evaporated. Gill says no oil should be passed that gives an evaporation of more than 4 per cent. with the above test. The United States Government requires that an oil should not lose more than 5 per cent. of its weight in 2 hours when exposed to 400° F.

RAILROAD SPECIFICATIONS AND STATE REQUIREMENTS

CHICAGO, MILWAUKEE, AND ST. PAUL RAILWAY COMPANY'S SPECIFICATIONS

41. Kerosene Oils.—These oils will be purchased subject to the following conditions and specifications: Dealers will be required to send to the purchasing agent a sample of each grade of oil they propose to furnish. Upon receipt of a carload of oil, one barrel will be selected at random and sampled. If this fails to pass all the following specifications of its class, the entire carload will be rejected and returned at the shipper's expense. If, after being accepted, any portion should be found damaged or inferior to the original sample, that carload will be rejected and returned as above. Packages must be strictly prime and in good order, with brand of oil and consignor's name stenciled on head with gauge marks, which will be subject to this company's verification. Oil containing glue or other foreign matter in suspension will be rejected. Oil that shows undue tendency to smoke or produce a white deposit on chimneys will be rejected.

42. Headlight oil, or so-called 150° fire-test oil, should have the following qualifications: (1) The oil must not flash below 110° F. when heated at the rate of 2° per minute, in closed-cup tester. (2) The gravity at 60° F. may vary between 46° and 50° Baumé. (3) The color must be clear, bright, and practically water white.

Winter oil must have a cold test not higher than 25° F. below zero, and is called *low cold-test oil.*

Spring and Fall oil must have a cold test not above zero, and is called *medium cold-test oil.*

43. Mineral seal oil, or so-called 300° fire-test oil, should have the following requirements: (1) The oil must not flash below 235° F. when heated at the rate of 2° per minute in closed-cup tester. (2) The gravity at 60° F. may vary

between 39° and 42° Baumé. (3) The color must be clear, practically white, with little or no bloom.

NOTE.—The Elliott closed-cup tester (standard in New York, New Jersey, and Iowa) is used in determining the above flashing points.

BALTIMORE AND OHIO RAILWAY SPECIFICATIONS

44. All oil is purchased by weight. Prices should be given in cents per pound (6.57 pounds per gallon). Shipments of one or more barrels which are filled with oil, cloudy from the presence of glue, or which contain dirt, water, or other impurities, will be rejected.

45. 150° fire-test oil (for office, switch, and station lamps) must have a flash test of at least 125° F. in a Tagliabue open cup, a fire test not below 150° F., and a cloud test not above 0° F. It must be water white in color and free from sulphur in any form; must be between 46° and 48° Baumé gravity at a temperature of 60° F.; must not flock when heated to a temperature of 270° F. for 1 hour, and must burn freely and steadily with the wick and burner used for this oil.

46. 300° fire-test oil (for lamps in passenger cars) must have a flash test above 250° F., a fire test not below 300° F., and a cloud test not above 32° F. When heated to a temperature of 425° F. and held there for 5 minutes, the oil must remain clear and transparent, showing but a slight darkening and no separation of flocculent or other matter—either at this temperature or on cooling. It must be *standard white* in color and free from sulphur in any form; must be between 38° and 42° Baumé gravity at a temperature of 60° F.; must weigh 6.85 pounds per gallon and must burn freely and steadily with the wick and burner used for this oil.

47. Signal Oil.—This grade of oil shall be prime white in color, shall contain not less than 40 per cent., by weight, of prime lard oil, and shall show a flashing point not below

200° F. and a burning point not above 300° F. The test will be made in an open vessel by heating the oil not less than 15° per minute, and applying the test flame every 7°, beginning at 193° F. When heated to a temperature of 450° F. and held there for 5 minutes, the oil must remain clear and transparent, showing but a slight darkening and no separation of flocculent or other matter—either at this temperature or on cooling. The gravity may be from 31° to 34° Baumé.

UNITED STATES QUARTERMASTER'S SPECIFICATIONS

48. **Mineral oil** to be water white, of a flash point not lower than 135° F. nor higher than 150° F. The specific gravity to be not less than 48° Baumé.

49. The **packages** to contain the oil to be cans of capacity of 5 gallons each, to be made throughout of IX bright charcoal tin, strictly first quality. Each can to have on top, near one corner, a 1¼-inch closed screw cap of uncut zinc or a screw cap with nozzle as made by the Record Manufacturing Company, of Conneaut, Ashtabula County, Ohio; or with Marsh faucets (11 Broadway, New York), each kind on half the number of cans. Weight of cans to be not less than 3 pounds each. Cases to contain two cans and to be made of ⅞-inch pine throughout, close jointed (dressed on outside), and of such size as to admit of the cans fitting closely on the sides. A thin, loose piece of board, full width and height of can, to be inserted between them to fit sufficiently close to hold the cans tightly in place. A strip of pine of proper thickness to be inserted between the cans and top of case, to protect the screw cap. The cans and cases to be made in the best workmanlike manner. The cases to be strongly nailed with not smaller than 8-pennyweight nails. Great care must be used in casing the oil that the nails are not driven into the oil cans.

50. The **oil** will be inspected and tested at point of delivery by sworn inspectors employed by the United States,

the instruments used being Tagliabue's pyrometer (closed cup) and Tagliabue's hydrometer.

51. The government inspectors heat the oil at 1° instead of 2° per minute in making these tests. Thus, the 135° F. requirement is about 128° F., as commonly taken on the Tagliabue closed tester.

COMPOUNDED OILS

52. First-Grade Cylinder Oil.—High-pressure cylinder oil for use in high-pressure cylinders: To be a mixture of the best petroleum stock and acidless animal oil, tallow oil being preferred. It must contain not less than 5 per cent. of pure, acidless animal oil, and have a flash point at not less than 550° F.; a fire test not below 600° F.; a cold test below 55° F.; and a gravity between 25½° and 27½° Baumé, at 60° F.

53. Second-Grade Cylinder Oil.—Low-pressure cylinder oil for use in low-pressure cylinders: To be a mixture of pure petroleum cylinder stock and acidless animal oil, tallow oil being preferred. It must contain not less than 10 per cent. of pure, acidless animal oil, and have a flash point of not less than 525° F.; a fire test not below 575° F.: a cold test below 40° F.; and a gravity between 26° and 28° Baumé at 60° F.

54. Machine Oil.—To be a free-running, hydrocarbon oil. May contain not more than 10 per cent. of acidless neatsfoot or tallow oil. The combined oil to be free from alkali or acid; flash point not less than 480° F.; gravity about 25° Baumé.

55. Dynamo Oil.—To be a free-running hydrocarbon oil. May contain not more than 5 per cent. of acidless neatsfoot or tallow oil. The combined oil to be free from alkali or acid; flash point not less than 402° F.; gravity about 28° Baumé.

56. The foregoing described oils must be free from all adulterations or mixture with animal, vegetable, or fish

oils, grease, lard, or tallow, except as stated, and contain no tarry or suspended matter, acid or alkali, glue or water.

METHODS OF TESTING THESE COMPOUNDED OILS

57. Flashing Point and Burning Tests.—Heat a small quantity of the oil in an open vessel, not less than 12° per minute, and apply the test flame every 10°, beginning at 250° F.

58. Precipitation Test for Tarry and Suspended Matter.—Mix 5 cubic centimeters of oil with 95 cubic centimeters of 88° gasoline, and if there is any precipitation in 10 minutes the oil will be rejected. This test is easiest made by putting 5 cubic centimeters of oil in a 100-cubic-centimeter graduate, then filling to the mark with gasoline and thoroughly shaking.

59. Test for Adulteration, Saponification, Etc.—This test depends on the property that mineral oils possess of not forming an emulsion when treated with alkali. Take 2 ounces of potassium hydrate, or of pure caustic soda, and dissolve in 8 ounces of distilled water. The sample of oil to be tested is put in a bottle, to which is then added at least an equal quantity of the potash or soda solution; shake well and allow to settle. Heating the mixture by plunging the bottle in a hot bath often assists this test. If the test is made in duplicate with a mineral oil of known purity, a comparison of the results obtained will be found valuable. Borax dissolved in water shaken into an equal quantity of mixed oil will be found to separate even small percentages of mineral oil. The borax solution must not be too strong, or a satisfactory result will not be obtained.

60. Test for Volatility.—When kept heated at 400° F. in an open vessel it must not lose more than 5 per cent. of its weight in 2 hours.

61. Test for Acid or Alkali.—It will be sufficient to wash a small quantity of the oil with distilled water; then drain off the water and test it with litmus paper.

TABLE V

STATE REQUIREMENTS

Alabama	No law.
Arizona	No law.
Arkansas	130° F. fire, Tagliabue open cup.
California	No law; cities, 110° F. flash.
Colorado	No law; cities, 110° F. flash.
Connecticut	110° F. fire, Tagliabue cup.
Delaware	110° F. fire, Tagliabue cup.
District of Columbia	120° F. flash.
Florida	130° F. fire, Tagliabue cup.
Georgia	120° F.
Idaho	No law.
Illinois	150° F. fire, Tagliabue.
Indiana	120° F. flash, Indiana cup.
Indian Territory	No law.
Iowa	105° F. flash, Elliott cup.
Kansas	110° F. fire, Tagliabue.
Kentucky	130° F. fire.
Louisiana	125° F. flash, Tagliabue.
Maine	120° F. flash, Tagliabue.
Maryland	No law.
Massachusetts	100° F. flash, Tagliabue open cup.
Michigan	120° F. flash, Foster cup.
Minnesota	110° F. flash, Minnesota cup.
Mississippi	No law.
Missouri	150° F. fire, Tagliabue.
Montana	110° F. flash.
Nebraska	Over 100° F. flash, Foster cup.
Nevada	No law.
New Hampshire	100° F. flash, Tagliabue.
New Jersey	100° F. flash, 115° F. fire.
New Mexico	150° F. fire.
New York	110° F. fire, Tagliabue.
North Carolina	100° F. flash, Foster cup.
North Dakota	100° F. flash.
Ohio	120° F. flash, Foster cup.
Oregon	No law.
Pennsylvania	110° F. fire.
Rhode Island	110° F. fire.
South Carolina	No law.
South Dakota	110° F. flash, Foster.
Tennessee	120° F. flash, open cup.
Texas	No law.
Vermont	110° F. fire, Tagliabue.
Virginia	110° F. fire, Tagliabue.
Washington	No law.
West Virginia	No law.
Wisconsin	120° F. flash, Wisconsin cup.
Wyoming	No law.

REQUIREMENTS

62. Requirements for Bidders.—Bidders must state specifically in their respective proposals the name and kind of each oil, its flash test, and specific gravity. Proposals, to receive consideration, must be accompanied by a certified check in the sum of five hundred dollars ($500), payable to the order of the Secretary of the Treasury, as a guarantee of good faith. The check of the successful bidder will be retained, with the understanding that the whole, or so much thereof as may be required, shall be used by the Department to supply any deficiencies that may arise owing to the inferior quality of the oils delivered, or delays in filling orders. Proposals must also be accompanied by samples of oils in 6-ounce vials, which must be labeled with the bidder's name and description of the oil, properly packed, and addressed to the Storekeeper, Treasury Department, Washington, D. C.

63. State Requirements.—Table V gives the requirements for illuminating or burning oils for forty-eight States and Territories. Table VI gives the requirements for foreign countries.

TABLE VI

REQUIREMENTS OF FOREIGN COUNTRIES

Belgium	35° C., Granier closed cup.
Denmark	40° C., Danish cup, open.
Germany	21° C., Abel closed cup.
France	35° C., Granier closed cup.
Japan	46° C. or 115° F., open cup.
Austria	21° C., Abel closed cup.
England	23° C. or 73° F., Abel closed cup.
Holland	40° C., Parrish closed cup.

64. Table VII gives the weight per gallon of petroleum and its products for different specific gravities.

TABLE VII

PETROLEUM AND ITS PRODUCTS

Degrees Baumé	Specific Gravity	Pounds Per Gallon	Degrees Baumé	Specific Gravity	Pounds Per Gallon
10	1.0000	8.33	49	.7821	6.52
11	.9929	8.27	50	.7777	6.48
12	.9859	8.21	51	.7734	6.44
13	.9790	8.16	52	.7692	6.41
14	.9722	8.10	53	.7650	6.37
15	.9655	8.04	54	.7608	6.34
16	.9589	7.99	55	.7567	6.30
17	.9523	7.93	56	.7526	6.27
18	.9459	7.88	57	.7486	6.24
19	.9395	7.83	58	.7446	6.20
20	.9333	7.78	59	.7407	6.17
21	.9271	7.72	60	.7368	6.14
22	.9210	7.67	61	.7329	6.11
23	.9150	7.62	62	.7290	6.07
24	.9090	7.57	63	.7253	6.04
25	.9032	7.53	64	.7216	6.01
26	.8974	7.48	65	.7179	5.98
27	.8917	7.43	66	.7142	5.95
28	.8860	7.38	67	.7106	5.92
29	.8805	7.34	68	.7070	5.89
30	.8750	7.29	69	.7035	5.86
31	.8695	7.24	70	.7000	5.83
32	.8641	7.20	71	.6990	5.80
33	.8588	7.15	72	.6956	5.78
34	.8536	7.11	73	.6923	5.75
35	.8484	7.07	74	.6889	5.72
36	.8433	7.03	75	.6829	5.69
37	.8383	6.98	76	.6823	5.66
38	.8333	6.94	77	.6789	5.63
39	.8284	6.90	78	.6756	5.60
40	.8235	6.86	79	.6722	5.58
41	.8187	6.82	80	.6666	5.55
42	.8139	6.78	81	.6656	5.52
43	.8092	6.74	82	.6619	5.50
44	.8045	6.70	83	.6583	5.48
45	.8000	6.66	84	.6547	5.45
46	.7954	6.63	85	.6511	5.42
47	.7909	6.59	90	.6363	5.30
48	.7865	6.55	95	.6222	5.18

WATER SOFTENING

(PART 1)

INTRODUCTION

WATER AND ITS PROPERTIES

1. Occurrence of Water.—Next to air, water is the most abundant and most important substance known. It occurs abundantly diffused in nature, both free and in combination, as sea-water, river water, spring or well water, in the form of clouds, fog, rain, snow, hail, and dew, as well as invisible vapor of water in the air, as a constituent of all plants and animals, and as water of crystallization in most minerals.

2. Composition of Water.—Water is composed of the elements hydrogen and oxygen, in the proportion of 1 volume of oxygen to 2 volumes of hydrogen. Water may be prepared synthetically by mixing appropriate volumes of hydrogen and oxygen in a suitable vessel and passing an electric spark through the mixture.

3. Properties of Water.—Absolutely pure water is a tasteless, odorless, and colorless liquid, although on looking through a layer of water 26 meters thick a pure dark-blue color may be observed. Being the most common liquid, water has naturally been taken as the standard for comparison of other liquids and solids. Its greatest density is at 39.2° F., or 4° C., 1 cubic centimeter at this temperature being the unit of the metric system of weights—the gram.

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Water becomes solid—ice—at 32° F., or 0° C., and a gas or vapor—steam—at 212° F., or 100° C. When absolutely pure it is slightly compressible, inelastic, and a non-conductor of electricity. Its solvent power is very great, and it may be said that absolutely or even approximately pure water is unknown as a natural product.

Its action has been one of the most important in producing geological changes. The solvent power of water, frequently increased by high temperatures and pressures, leaches out soluble minerals and salts from strata, and through the deposition of these materials under varying conditions, new minerals and changes in strata are produced. On account of its almost universal solvent power on the majority of rocks and soils, or on some of their constituents, pure water is unknown in nature. Even rainwater before coming in contact with the earth washes impurities out of the air. Usually, these impurities consist of gases, and in many instances suspended matters also.

4. As just stated, pure water is not met with in nature, at least for industrial purposes; and, indeed, absolutely pure water is unknown naturally. The term *pure water* is used here in a relative sense only, meaning thereby a water containing a comparatively small amount of impurities or a quantity not injurious for the special purpose, whether it be a potable water or one that is to be used industrially. The nearest approach to a pure water in nature is rainwater, and this is only approximately, but amply so for all industrial purposes, although the dissolved gases are believed to cause pitting and corrosion in steam boilers. Rainwater in falling, as already stated, washes out and absorbs impurities, mostly gases; then, on being collected from roofs, the mechanical impurities, some of which are partly soluble, render it still more impure. Rainwater, however, is directly of no practical importance as an industrial supply, its use being confined to domestic purposes, which in many localities are quite extensive and consequently represent a large factor in domestic economy.

5. In whatever form it reaches the earth—as rain or as snow—water may contain the gases of the air to a very great extent. Carbon dioxide, CO_2, may be dissolved up to nearly 1 per cent. of the volume of the water; and air itself up to 5 per cent. Nitric acid or oxide, and ammonia, NH_3, are frequently present, being washed out of the atmosphere and absorbed under certain meteorological conditions. The oxygen absorbed causes the water to have slightly oxidizing properties, and the presence of carbonic-acid gas gives it the property of a weak acid. Many rocks and minerals are readily dissolved and leached out, producing waters highly charged with the soluble constituents. The alkali chlorides, sulphates, and carbonates especially pass readily into solution.

6. Action on Rocks of Water Containing Carbon Dioxide.—The action of water on many rocks and minerals is greatly increased by the presence of absorbed carbon dioxide. This is especially true of certain carbonates that are only slightly soluble in pure water. The most important of these carbonates are the minerals *limestone*, *dolomite*, *magnesite*, and *siderite*. In addition to these compounds, which may be considered to go directly into solution, there are other apparently insoluble compounds that are more or less affected by the water containing carbon dioxide. These go into solution as carbonates and bicarbonates.

As the water descends into the earth, more carbon dioxide is absorbed from the oxidation of organic matter and the breaking down of mineral carbonates, and the solvent power of the water is thereby increased.

7. Action of Atmosphere and Water on Rocks. Under the influence of the atmosphere and running water, rocks are loosened, broken up, and more or less worn away. This action is partly chemical and partly mechanical, but the most energetic is that resulting from the breaking up of rocks by freezing and thawing. Water at 0° C., or 32° F., expands to nearly 1.09 times the original volume of the water on freezing, the specific gravity of ice being .9. In consequence of this expansion, an enormous bursting pres-

sure is exerted and immense masses of rock are broken up. Soluble components are thus rendered open to the action of the water and pass into solution.

8. Reactions That Take Place in Waters.—Waters that are naturally soft come from formations that are nearly insoluble, such as granite, the pure clays, gneiss, etc. Many waters having their source in such deposits, however, flow over or through lime formations, as calcium or magnesium carbonate or sulphate, and take these up, as already stated. These constitute the hard waters, and both their origin and most of their course may be in such a region.

Furthermore, it may be said that a salt or a compound once in solution may be changed to another by a variety of reactions occurring with matter taken up at a later stage. For example, calcium sulphate (gypsum) and magnesium carbonate gradually change to magnesium sulphate and calcium carbonate, the latter passing out of solution in the absence of carbonic-acid gas; but generally calcium carbonate will be present, and the lime will go into solution as bicarbonate. If sulphur is present as an organic compound, or more commonly as pyrites, sulphuric acid or sulphate of iron may result. In a similar manner, a great many reactions occur, forming new salts or compounds differing from those originally taken into solution.

9. Hardness of Water.—As has been shown, almost all river and spring waters hold more or less mineral matter in solution, and owing to the prevalence of limestone formation, lime in the form of calcium sulphate or as an acid carbonate, as well as magnesium, is always present in such waters. A water containing calcium and magnesium compounds in solution forms very little, if any, lather with soap, and is termed **hard water** in contrast to a water free or nearly free from those compounds, which is called **soft water.** If the hardness is due to acid carbonates, generally known as calcium and magnesium bicarbonates, it disappears on boiling, the calcium and magnesium bicarbonates being decomposed and precipitated as calcium and magnesium

carbonates. In such a case, the water is said to possess merely *temporary hardness*. In boilers, the precipitated carbonates of calcium and magnesium cling firmly to the inner surface of the vessel and form what is known as *boiler scale*. If the hardness is due to sulphates that are only partly removed by boiling, as, for instance, calcium sulphate, it is spoken of as *permanent hardness*.

EARLY WATER-SOFTENING METHODS

10. Original Water-Softening Process.—The method of water softening in use today depends on a process patented in 1841 by Dr. Thomas Clark, of Aberdeen, Scotland. It consisted in using caustic lime or lime water to soften hard water, and his patent covered apparatus for the purpose. The object was successfully accomplished with waters of a certain class, but compared with the apparatus in use today, his was essentially crude. The cost of treatment was also prohibitive, except in cases of the hardest waters.

Prior to this method, however, in 1766, Cavendish discovered that lime water added to certain hard waters would soften them by throwing out of solution the carbonates of lime and magnesia. Thomas Henry in the latter part of the 18th century proposed to soften water in the same way, but Clark was the first to apply the process practically. Clark also developed certain tests that remain in use today with less modifications than most chemical methods after three-quarters of a century's use. These tests are the alkalinity test for ascertaining the amount of lime required to soften a given water, and the silver-nitrate test, which determines whether an excess or a deficiency of lime has been used, so that the treatment may be corrected accordingly. Clark is also generally credited with suggesting the soap test—the use of a standard soap solution for determining the hardness of water. These tests are of the greatest value as a control in water-softening operations, especially as their application is easily acquired and their results reliable and, within certain limits, usually sufficiently close for practical purposes.

For waters having their hardness due to carbonates of lime and magnesium, Clark's original process was essentially perfect from a chemical standpoint; but it did not soften waters having their hardness caused by other compounds.

Mechanically, the apparatus was crude. It consisted of masonry tanks built in the ground for the lime water, for mixing and reaction, and for settling. The settling required from 12 to 24 hours, thus necessitating an extremely large tank capacity for any considerable volume of water—a factor that made the cost prohibitive in many cases.

11. Caustic-Lime-and-Sodium-Carbonate Process. There is some confusion as to who first proposed and applied the process in which caustic lime and sodium carbonate are used in conjunction. This process was an extension of the original Clark process, which merely removed the carbonates. The use of the two reagents simultaneously, or of sodium carbonate after the caustic lime had partly or completely removed the carbonates, transformed the sulphates as well, was considered a great advancement in the softening of water. The process is generally credited to Porter, but F. Schulze suggested the use of these reagents in Germany, in 1876, and Archbutt and Deely also proposed their use about the same time. There is not much definite information available as to what extent the original apparatus was applied.

The process consisted in treating the water with a solution of lime and soda, or both; thus, it was more a question of the apparatus than of the chemical treatment. The treating solution was forced into a mixing tank by pressure, and after becoming thoroughly incorporated with the raw water, and sufficient time allowed for the reactions, it was forced through a filter press. The scheme was very ingenious, but it had several serious drawbacks. Among them were high initial cost, heavy operating expense, and constant attention required to keep the feed of chemicals regular.

It will thus be seen that while the principles and chemical reactions involved in water softening have been known for

a long time, the development of softening apparatus has been of comparatively recent date. Little change seems to have been made from the original apparatus of Clark until some 25 years ago. Beginning about 1880, many forms of softening appliances were brought out in Europe. These appliances were later adopted in America, where many important modifications were brought about.

12. Disadvantages of the Early Methods.—For a number of years the tendency was to confine the treatment to the water in the boilers or in preheaters, immediately before entering the boiler; and in locomotive practice, the reagents were generally added to the engine tank. By this practice, the precipitated matter, or sludge, was not removed before entering the boiler. Consequently, the efficiency of the process was greatly reduced, as part of this precipitate would either form scale or require frequent blowing off to remove the sludge and thus prevent foaming in the boiler.

While the method of treating the water in the boiler gave greatly improved results, the present method of softening the water outside the boiler and then removing all sludge by filtration or sedimentation, is superior and more economical.

13. Comparison of Intermittent and Continuous Systems.—All the early apparatus was of the intermittent type. Usually, only two tanks, together with suitable tanks for the solution of the reagents and their addition, were employed. Very early in the period of development previously referred to, the advantages of a continuous softening process were recognized, and many efforts were made toward this end. Many of the problems involved have taken quite a number of years to be successfully developed, and the work of many inventors was required to perfect them.

The intermittent system, with sufficient intervals for the reactions to take place and for the precipitated matter, or sludge, to be removed by sedimentation or filtration, or, as is commonly the case, by a combination of the two, is comparatively simple from a mechanical standpoint, while the continuous system offers more serious mechanical problems.

Even today, the advantage of the former over the latter system is perhaps due to this fact.

As the quality of the water supply in the intermittent system was regular or was changed at short intervals, fixed or varying amounts of reagents could easily be weighed out and added to a known amount of raw water. Nothing more certain and exact could be desired. But many advantages

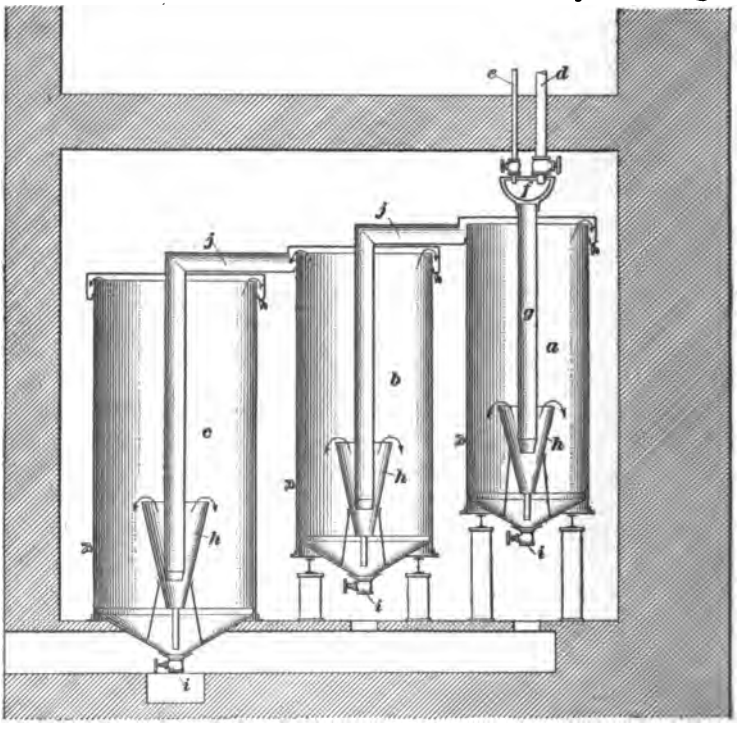


FIG. 1

belonging to a continuous scheme were apparent; as, for example, the much smaller space required to install a plant of a given capacity, a continuous regular supply of soft water, and less expense of installation and operation.

The chief difficulties encountered in perfecting the continuous system were to proportion accurately the amount of chemicals to be added and to adjust the additions either to a

change in the rate of flow of the water or to a change in its quality.

These difficulties have been overcome and are now taken care of accurately and efficiently by means of apparatus. This granted, there would appear at first sight to be no question as to the relative desirability of the two types. Yet it remains that the simplest and the best designed mechanisms are subject to disarrangement, accidents, and renewals. On these points, the intermittent system necessarily maintains its superiority, and today both systems are being installed, either special local conditions or the cost of installation usually determining whether it shall be the one or the other system.

14. Earliest Type of Continuous Apparatus.—It appears that the German and French chemists and engineers led in the development of continuous water softeners, and that Berenger and Stingl installed the first continuous softening plant.

The earliest form of apparatus employed by Berenger and Stingl is shown in Fig. 1. It consists of three main tanks *a*, *b*, and *c* that are connected with each other, each succeeding tank being placed on a slightly lower level. The raw water enters through the pipe *d* and the reagent through the pipe *e* into the mixing vessel *f*. Both pipes are supplied with cocks to control and proportion the flow of both the reagent and the raw water. The mixture consisting of the reagent and the raw water is allowed to run through the pipe *g* into the cone-shaped cup *h*. From this cup the mixture overflows into the main tank, and in doing so it deposits part of the sludge, which settles in the conical bottom. The water gradually rises in the tank, depositing the sludge as it ascends. When the water reaches the top of the tank *a*, it is carried by pipe *j* into the cup *h* of tank *b*, and so on, until it finally reaches tank *c*, to the top of which a filter of wood fiber or some similar material is sometimes attached. Each tank has a valve *i* in the conical bottom to discharge accumulated sludge into the sewer.

15. While the arrangement of the apparatus is simple and the apparatus itself comparatively cheap, not frequently requiring costly repairs, its success was impaired by the fact that it was practically impossible to control and proportion accurately the flow of reagent and water, thus lacking the accurate adjustment necessary for a water varying in quality.

On the other hand, the wood-fiber filter through which the upward current of water passes from the softening apparatus to a storage is a feature used in some of the most recent and popular types of continuous apparatus. The most important feature of the apparatus, however, was the principle of change in direction of the flow and its diminished velocity as it emerged from the pipe *g*, Fig. 1, to the entire area of the tank. This principle, applied in various ways, is still a feature of a large number of continuous apparatuses.

While the original apparatus had but a limited use, it is important to consider the development of this type of apparatus, as some of its points were carried along in later devices and are plainly in evidence today. The original inventors later made various improvements, but their apparatus remained essentially the same as the early form of it. Its comparative failure was probably due to the improper mixing of reagents with raw water and the subsequent reaction being imperfect, rather than to the faulty design of the device proper.

WATER PURIFICATION AND ITS BENEFITS

GENERAL REMARKS

16. To those in intimate touch with water consumption on a large scale, especially where the scale-forming, corrosive, or soap-destroying properties of water are in daily evidence, no argument is required to show the necessity of rectifying these troubles or the economy resulting from such rectifications. Considering, however, that Clark proposed the water treatment in 1841 and that nearly 40 years had elapsed before such processes were generally taken up and used in Europe, and, besides, that the treatment of water for industrial purposes in the United States only dates back to 1892, except in isolated cases, some explanation of the needs of such treatment and the benefits derived therefrom does not seem amiss.

17. Purposes of Water Treatment.—Waters to be used for commercial purposes are treated for one or more of the following reasons: (1) To remove scale-forming matter held in solution; (2) to neutralize or remove corroding substances; and (3) to remove suspended matter, such as mud, sand, etc. All three of these are usually accomplished in one operation, the first two by chemical means, through the addition of definite amounts of suitable materials; then, by the mechanical removal (by filtration or sedimentation) of the sludge, or precipitate, resulting from this reaction, any clay, sand, etc. present is removed along with the sludge.

BOILER SCALE

18. Definition of Scale.—The incrustation that forms in or on boiler tubes and sheets, due to deposits from the feedwater, is called **scale.** It consists essentially of calcium deposits that vary greatly in composition according to the character of the water evaporated. Not all substances in solution form scale; some of them settle out as the water evaporates. Those which form scale will be taken up later.

19. Disadvantages of Boiler Scale.—The deposit of scale is highly non-conductive of the heat applied to raise

TABLE I

LOSS OF HEATING POWER DUE TO BOILER SCALE

Thickness of Scale Inch	Loss of Heating Power or Waste of Fuel Per Cent.	Thickness of Scale Inch	Loss of Heating Power or Waste of Fuel Per Cent.
$\frac{1}{64}$	2	$\frac{1}{4}$	38
$\frac{1}{32}$	4	$\frac{3}{8}$	48
$\frac{1}{16}$	9	$\frac{1}{2}$	60
$\frac{1}{8}$	18	$\frac{5}{8}$	74
$\frac{3}{16}$	27	$\frac{3}{4}$	90

steam, thereby greatly increasing the cost of fuel. These scale deposits vary in thickness from a barely perceptible, easily removed deposit to one that completely fills up boiler tubes. The character of the deposits differ as widely, varying from soft pulverulent deposits or soft friable ones to those of a hard, crystalline character. The latter deposits sometimes adhere to the tubes and shell so firmly that a hammer and chisel or special boiler-cleaning tools to bore out the tubes may be required. Cleaning with such instruments is expensive, and may result in injury to the boiler. Another loss is caused by the boiler being out of service. In such cases, the plant is deprived of power or the boiler equipment for a given horsepower must be increased. What

actually happens, as a rule, is that the remainder of the boiler plant is driven beyond its normal, and sometimes its safe, capacity while boilers are being scaled. In this way, not only is the cost of maintenance of the boiler plant greatly increased, but its possible and proper life is greatly decreased.

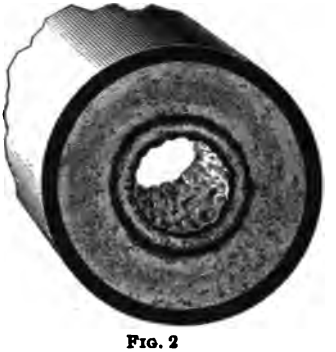


FIG. 2

20. Loss of Heating Power Due to Boiler Scale. According to different authorities, the loss of heating power or the increased amount of fuel required for a given thickness of scale varies somewhat. But all agree that there is a considerable loss occasioned by a coating of scale on the tubes or sheets of a boiler. This discrepancy is probably due to the varying physical properties and chemical composition of the boiler scale.

Table I shows the loss of heating power due to boiler scale of various thickness. While the figures given are considered to be conservative and authoritative, it should be stated that such extreme losses are seldom met with in practice.

21. Examples of Scale Deposits.—In order to illustrate the effect of using very hard waters in boilers, a section of a tube in which there is a deposit of scale is shown in Fig. 2. The scale last deposited in the center is less dense than the portion nearest the shell of the tube. The reason for this is that the former has not lost all its moisture and possibly volatile matter, while the latter has been baked to a hard, compact deposit. The condition illustrated is by no means exaggerated, and may be found in boilers that have not been frequently cleaned. The loss occasioned by such deposits is enormous.

22. Fig. 3 shows a number of views of scale deposits and flues taken out of locomotive boilers that had used

different kinds of water. These exhibits show the effects of hard waters, and a study of them will give a definite idea of the consequences of using hard waters.

In (*a*) is shown a soft, bulky scale deposited from a water carrying calcium carbonate only, and in (*b*), a hard, thick

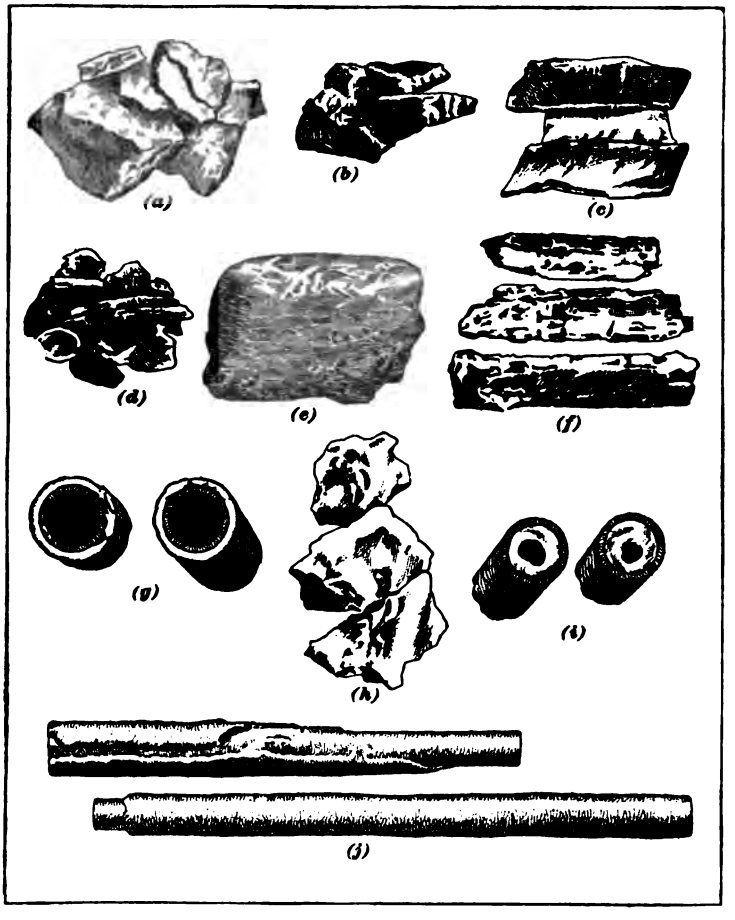


FIG. 8

scale from a water containing calcium sulphate; some of this was $1\frac{1}{4}$ inches thick. The scale shown in (*c*) is $\frac{3}{8}$ inch thick and was taken from the outside of locomotive flues. View

(*d*) shows small pieces of soft scale removed from locomotives by the means of treated water. The cup-shaped pieces were deposited over rivet heads, and the cylindrical ones around staybolts. In (*e*) is shown a large chunk of scale removed from a boiler, and in (*f*), samples of scale deposited around flues; some of the latter pieces were $\frac{7}{8}$ inch thick. In (*g*) is shown pieces of flues coated with a hard, dense scale from $\frac{1}{4}$ to $\frac{5}{8}$ inch thick, and in (*h*), a sample of the dried white precipitate removed from a carbonate water by softening. This precipitate is a relatively pure carbonate of lime, with an admixture of clay and some of the other impurities contained in the raw water.

Locomotive feedpipes, nearly filled with scale, are shown in (*i*). In these examples, the openings for water are reduced to $\frac{1}{4}$ and $\frac{1}{2}$ inch. The deposit is from a carbonate water and is due to the heat driving out the carbonic acid in the water that held the calcium carbonate in solution. In (*j*) are shown long pieces of flues covered with scale from a sulphate water. On the longer piece, the scale was so firmly united to the flue that a hammer and chisel were required to remove it.

23. Conductivity of Scale.—It is generally stated that the average conductivity of boiler scale is one-thirty-seventh that of iron. This is probably not accurate, as the relative non-conductivity compared with iron increases much faster than the thickness of the scale. Waters that are exclusively carbonate deposit either a loose, brittle scale or a soft sludge practically all of which may be removed by blowing out. A scale of this character is the exception rather than the rule, and those usually met with contain enough sulphate to cement the scale quite securely to the tubes or sheets. Such scales vary from those easily removed by the usual cleaning or scaling tools to one that is so attached to the metal as to be almost an incorporated part of it. The scale from waters high in sulphate show the latter character, being themselves mainly composed of sulphates. If composed exclusively of sulphates, the scale is hard, white, crystalline, and of great beauty, and

if it is broken off from the metal, corrosion of considerable extent and a blackish paste next to the metal will usually be shown. This paste is due in all probability to a reaction between the calcium sulphate of the scale and the metal of

TABLE II

COST OF OPERATING A BOILER PLANT

USING UNTREATED WATER

Month	Fuel	Cleaning	Repairs	Incidentals	Operating Labor
January	$ 2,271.50	$141.00	$ 43.00	$ 18.00	$ 90.00
February	2,563.00	172.40		12.80	90.00
March	2,496.00	162.34	96.50	16.20	90.00
April	2,371.90	65.00		11.00	90.00
May	2,640.00	176.50		19.30	90.00
June	2,376.00	147.20	49.00	23.28	90.00
Total	$14,718.40	$864.44	$188.50	$100.58	$540.00

USING TREATED WATER

Month	Fuel	Cleaning	Repairs	Incidentals	Operating Labor
January	$ 2,163.00	$23.00	$94.00	$12.25	$ 90.00
February	2,241.00	17.00		19.30	90.00
March	2,164.00			6.50	90.00
April	2,196.00			16.95	90.00
May	2,230.75			23.80	90.00
June	2,084.00			13.40	90.00
Total	$13,078.75	$40.00	$94.00	$92.20	$540.00

the boiler forming sulphate of iron, which, owing to the high temperatures reached by the shell or tubes when covered with such a scale, is either decomposed into sulphuric acid or the salt acts directly on the metal. With waters having more

carbonates than sulphates, the scale formed in the boiler is not so hard and compact, and it may be removed much more readily with scaling tools. Since such scale is less dense and cohesive, it is less of a non-conductor than the dense sulphate scales; hence, it causes less waste of fuel than the sulphate scales for the same thickness.

24. Economical Results Obtained From Water Treatment.—In Table II is given a practical illustration of the economy resulting from correcting the evil effects produced by bad water. The principal items of expense in operating a boiler plant of moderate size for 6 months before the installation of a water-softening plant are given in the first part of the table, and in the second part is given the cost of operation for the 6 months succeeding the installation.

25. According to the totals given in Table II, the difference in cost of using treated water instead of untreated water is as follows: Fuel, $1,639.65; cleaning, $824.44; repairs, $94.50; and incidentals, $8.38, making a total for 6 months of $2,566.97. By deducting from this total $487.50, which will cover the interest on the original cost and the depreciation of the plant, the net saving will be found to amount to $2,079.47. The cost to install the water-softening plant was about $4,000. Therefore, there was a saving of practically 50 per cent. on the outlay in 6 months. The principal items to show a marked saving are fuel, cleaning, and repairs, the savings on these items amounting to about 11, 95, and 50 per cent., respectively.

The results just shown are by no means phenomenal, but are perhaps greater than the average results obtained. However, a saving in the operation of steam plants of from 40 to 50 per cent. per year on the cost of the softening plant is very ordinary, and is reached in most instances if a proper preliminary study of conditions has been made. Each installation is a special case, and owing to various local conditions, waters that are similar may give varying figures as to the final economy. It seldom pays to soften a water

TABLE III

COMPOSITION OF BOILER SCALE FROM DIFFERENT WATERS

Scale No.	Source of Water	$CaCO_3$ Per Cent.	$CaSO_4$ Per Cent.	$Mg(OH)_2$ Per Cent.	$MgCO_3$ Per Cent.	Sodium Salts Per Cent.	Fe_2O_3 Al_2O_3 Per Cent.	SiO_2 Per Cent.	Moisture and Organic Matter Per Cent.
1	River and well water	4.07	83.83	2.78					
2	River and well water	17.79	63.62	8.50					
3	River and well water	85.98	6.89	4.05					
4	River water	75.85	3.68	2.56		.45	2.96	7.66	6.84
5	River water	75.92	3.16		10.16	.84	2.96	4.94	2.02
6	River water	81.45	1.63	8.10			2.44	.87	5.51
7	River water	8.20	85.01	4.36			.52	1.91	
8	River water	59.38	7.09	16.35			8.29	9.00	
9	River water	51.43	4.44	19.43			11.64	12.85	
10	River water	50.04	1.28	21.47			16.51	10.90	
11	Well water	32.16	5.64		20.04	3.31	7.46	16.94	14.45
12	Well water	25.62	55.92		5.56	.22	5.04	5.26	2.38
13	Town supply	62.95	20.80		7.24	.86	2.48	3.76	1.91
14	Town supply	50.04	29.76		10.84	.86	2.36	4.28	1.86
15	Town supply	17.31	53.76		18.04	.54	2.88	4.36	3.11
16	Town supply	1.22	78.32		10.36	.64	4.64	3.22	1.60
17	Sea-water	2.00	34.00	58.00		Trace	1.33	Trace	4.67
18	Sea-water	3.44	69.77	22.50		.99	1.36	.16	1.78
19	Sea-water	.34	72.85	18.83		2.16	2.40	.80	2.62
20	Sea-water	.97	85.53	3.39		2.79	.32	1.10	5.90

below a hardness of 6° or 7° (6 or 7 grains of incrusting matter per United States gallon), and the greater the hardness above this, the more economy a water-softening installation will show.

26. Composition of Boiler Scales.—In Table III are given analyses of scales of varying compositions.

27. Characteristics of Scales and Waters Producing Them.—Referring to Table III, the scales numbered 1, 2, and 3 are from locomotive boiler tubes. They represent deposits ranging from an extremely hard scale, No. 1, to a spongy, friable, coarsely crystalline scale, No. 3. Scale No. 2 is between these two in hardness, but is nearer to No. 1, as it contains enough calcium sulphate to give a very hard scale; the large amount of magnesia, $Mg(OH)_2$, increases the hardness also. While complete analyses of these three scales might be desirable, the three constituents account for over 90 per cent. and will serve the purpose here as well as a full analysis; the remainder would be principally silica, oxide of iron, and alumina. The analyses of the waters from which these deposits were formed show an interesting comparison of the scale formed from a given water. The analyses, in parts per hundred thousand, are as follows:

	$CaCO_3$	$MgCO_3$	$CaSO_4$	$MgSO_4$	$MgCl_2$
No. 1 . . .	5.87	.78	4.61	2.37	.26
No. 2 . . .	11.91	1.50	1.63	2.93	
No. 3 . . .	6.43	.85	.87	.29	.59

The waters show increasing proportionate amounts of carbonates to sulphates, and a corresponding increase of carbonate is shown in the scale.

28. The scales numbered 4, 5, and 6 were formed from waters that were high in temporary hardness (carbonates) and low in sulphates. No. 7 was a water high in sulphates. Nos. 8, 9, and 10 were scales from a highly acid river water that had been treated by lime and soda ash to neutralize the acid and reduce the permanent hardness. They were soft, friable scales, and indicate a good degree of softening. The

water was high in ferrous sulphate, which accounts to some extent for the high oxides of iron and alumina.

Nos. 11 and 12 were produced from well waters, the former being a magnesian water, as is shown by the high amount of magnesium carbonate in the scale. It is unusually high in silica, and the high percentage of oxide of iron indicates that there was perhaps action on the metal of the boiler owing to the presence of some corroding substance—probably magnesium chloride. No. 12 was mainly a sulphate water, but it was also high in temporary hardness (carbonates).

Nos. 13, 14, 15, and 16 indicate the class of waters from which such scales would deposit, as shown by preceding scales; that is, whether river or well water, or both, in varying proportions are used.

Nos. 17, 18, 19, and 20 are scales from sea-water, and while such feedwater is seldom used exclusively, even on ocean steamships, it frequently has to be used partially. These scales were formed under increasing pressures, and are of special interest as they show the increasing proportion of calcium sulphate precipitated at higher pressures and temperatures (less calcium sulphate being held in solution with increased temperature). Stillman gives the following as the range of composition of various ocean waters:

Substance	Parts per Hundred Thousand
Ca	32.4 to 55.7
Mg	115.8 to 147.1
Na	1,020.5 to 1,177.0
K	35.4 to 66.8
SO_4	259.0 to 303.0
Cl	1,816.8 to 2,084.0
Br	23.9 to 38.8

29. Attention is called to the fact that while the analyses of a number of the preceding scales show magnesium carbonate, magnesium hydrate usually is found to the exclusion of the carbonate. This may be due either to a wrong interpretation of the analyses and an improper combination of

bases and radicals, or the deposits may have absorbed carbonic acid, CO_2, on exposure to the air.

30. In addition to the economies just outlined, the softening of water for boiler purposes does add a large safety factor to the operation of a boiler plant. Heavy scale deteriorates the boiler, not only in efficiency but in safety. The incrustations being non-conductive and irregularly distributed, may lead to local overheating. At times, portions of the boiler in direct contact with the flames may be heated to redness, owing to the fact that a dense and thick scale is deposited that prevents the water from absorbing the heat and circulating it evenly throughout the boiler.

At such times, that part of the boiler is subject to sudden expansion, which may cause the cracking of the scale and allow the water to reach the overheated metal, thereby producing a sudden or even explosive generation of a large volume of steam. The results may not be more serious than the loosening of rivets or the opening of seams; but, on the other hand, the heating may cause a tube or sheet to burst, and even serious explosions may result.

CORROSION AND FOAMING

CORROSION

31. Causes of Corrosion.—A trouble that is even more acute than the formation of scale is **corrosion,** which pits and eats sheets and tubes, and with many waters very rapidly puts the boiler out of service. Many natural waters are more or less corrosive owing to the presence of dissolved carbonic-acid gas, oxygen, certain salts, or free acids. Rainwater containing no salts but merely gases in solution is corrosive under boiler conditions. In such cases, it is believed that the corrosion is due mainly to the dissolved oxygen.

Iron will not corrode in pure water, but the presence or gradual absorption of carbonic-acid gas or oxygen will start corrosion under ordinary conditions, and at the temperature and pressure of the boiler this corrosive action greatly increases. As iron ordinarily rusts, ferrous carbonate is first formed; this then dissolves in the water by the aid of additional carbon dioxide, forming bicarbonate of iron. The bicarbonate of iron is finally transformed into hydrated sesquioxide, after passing through the state of magnetic oxide, through the agency of the air and water.

Waters may be both corrosive and scale forming. Usually, if a water has a tendency to corrode, the corrosion is invariably accompanied to a greater or less extent by scale formation. Such water is commonly known as *incrusto-corrosive water.*

32. Treatment of Corrosive Waters.—Corrosive waters may be treated in the boiler direct by adding a neutralizing agent, such as caustic or carbonate alkalies, or caustic lime. This matter has to be handled with intelligence and care, or the scale-forming matter may be greatly increased. A water containing sulphuric acid or ferrous sulphate neutralized with lime will form calcium sulphate, which is the worst possible incrustive salt. Treatment outside the boiler, the same as for the removal of incrusting salts, is the only rational course and the most economical in the end.

33. Action of Corrosive Waters on Steel and Wrought Iron.—In connection with corrosion of boilers in general, it may be said that the more mildly corrosive waters usually attack steel boiler shells and tubes somewhat more vigorously than those made of wrought iron. Without presuming to advance any definite or positive statement in explanation, as the matter is considerably in dispute, the general statement that the wrought iron is less susceptible to attack than steel seems to be well borne out by general experience. No exact explanation is widely accepted, but the general idea seems to be that the fibrous nature of the

wrought iron, together with the partial protection given the metal fibers by the presence of the slag always contained in wrought iron, is a large and very important factor in the greater resistance offered to corrosion. Furthermore, the almost complete absence of manganese in wrought iron is a factor in retarding this disintegration. The increased corrosion that occurs when manganese is present is probably due to galvanic action set up in the case of steel. The presence of nickel, chromium, and cobalt retards corrosive action, but none of these alloy steels has been used to a very great extent in boiler construction. Regarding the relative resistance of iron and steel to corrosion, it should be said that if a water containing sufficient sulphuric or

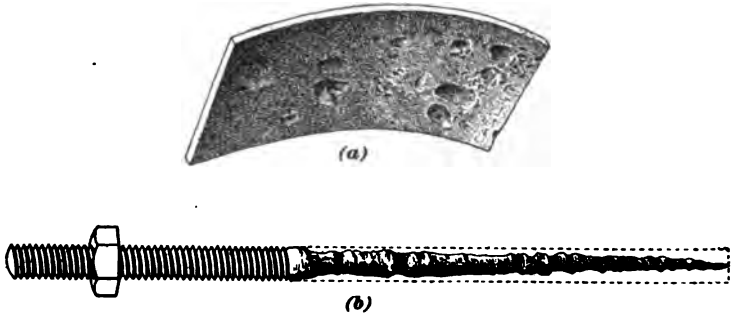


FIG. 4

hydrochloric acid or corrosive chloride to form a free acid is used, no great difference will be found between steel or wrought iron; but if the water causes less acute and extreme corrosion, a difference will generally exist in favor of the wrought iron.

34. Examples of Corrosion.—Corrosion is usually confined to certain parts of the boiler, but it may be quite general. Whether localized or general, it depends on the nature of the corroding substance or substances in the water, the type of boiler, and the manner in which the boiler is worked.

Corrosion may be shown as pittings over a more or less extended area. The pits, or cavities, are usually approxi-

mately round or spherical, cup-shaped holes, and are generally filled with a black powder or sludge composed mainly of hydrated oxide of iron, together with the corroding salt or the corresponding iron salt resulting from the reaction with the metal. These cavities gradually increase in depth until the weakened metal opens under boiler pressure.

In Fig. 4 (*a*) is shown a case of pitting caused by the action of free acid in the water or by salts yielding acids on decomposition. These pits may be merely superficial, or they may have so penetrated the shell or tubes that only a mere sheet of metal remains. Fig. 4 (*b*) shows a bolt that has been nearly destroyed by acid corrosion. Fig. 5 shows a case of general corrosion over a considerable area of a sheet. Both forms of corrosion may become acute, and are sources of loss owing to the more or less extensive repairs that must be made, to say nothing of the danger of accident.



FIG. 5

35. Effect of Mechanical Action on Corrosion.—Corrosion may be greatly increased by **mechanical action.** Owing to varying pressures on different parts of the boiler, plates and sheets bulge and seams open, allowing the water or steam to leak out in jets. This greatly increases the corrosion, or, more correctly speaking, creates an abrasive action, producing a local effect that is very similar to corrosion and is sometimes mistaken for it. Poor workmanship in boiler or steam-pipe construction may develop the same condition.

FOAMING

36. A trouble that occurs occasionally with raw water is **foaming, or priming.** This trouble, however, also occurs with treated water, if the salts in solution are allowed to reach too great a concentration in the boiler, or if the water naturally contains such an amount of salts that the softening reactions produce an excessive amount of soluble salts. This foaming, or frothing, is generally attributed to highly alkaline waters, but it has been demonstrated that the alkalinity has much less to do with foaming than is commonly assigned to it. While alkaline waters frequently foam badly, this alkalinity, by itself, is not the cause of this trouble. The real cause is nothing more than too great a concentration of the water, or, in extreme cases, soluble salts or other matter in suspension due to supersaturation of the water in the boiler. Oily substances introduced into the boiler cause foaming for the same reason.

37. The terms *foaming* and *priming* are generally used synonymously, but the latter term should be used to indicate a more aggravated form of the trouble. The effects of foaming and priming vary from wet steam with more or less vapor up to the point when water is thrown up into the steam space and may thus be carried over with the steam to the engine cylinder, always with the danger of bursting out cylinder heads and producing serious accidents. Aside from this, the smaller amounts of water carried over in the steam as vapor absorb an amount of heat that may represent no inconsiderable loss in boiler economy.

The trouble of foaming or priming may be checked by temporarily shutting down the boiler until the water subsides or by withdrawing or checking the fire until the violent ebullition ceases. This relief is only momentary, and with hard firing will likely recur immediately. The concentrated water containing suspended or insoluble matter should be partly removed by blowing off and then replaced by fresh water. As the degree of concentration constantly increases, many waters will not be sufficiently remedied by blowing

off, and at intervals the boilers will have to be washed out and all the old water removed. The limit of relief afforded by these measures depends on the character of the water, the degree the boiler is driven, and the construction of the boiler, as boilers with insufficient steam space foam worst.

38. Authorities do not seem to agree in regard to the amount of salts in a given quantity of water that will produce foaming, and no hard-and-fast rules can be laid down, as other factors have to be taken into consideration, namely: (1) precipitated sludge; (2) silt or other suspended matter carried by the boiler feedwater; (3) organic matter present in the water; and (4) scale loosened from shell or tubes and circulating in the boiler. All these and perhaps other factors influence foaming, from which can readily be seen the difficulty of determining the amount of salts in solution that produce it.

Foaming may accompany a certain amount of alkali or other salts and yet be largely influenced by one or more of the preceding factors, when it could not be properly said that such an amount of salts alone produced the foaming. Under working boiler conditions, it is next to impossible to gauge with any degree of accuracy the influence of these other conditions; hence, the error liable to arise in stating that a certain percentage of salts in the water caused foaming.

39. It is variously stated that waters containing from 150 to 250 grains of alkali salt per gallon will cause foaming, and that half this amount produces foaming in the presence of suspended matter. From this statement it must not be taken that the preceding amount of alkali salts would be permissible in feedwater, as it would then stand no concentration whatever, but would foam at once when steam was raised. From the causes just given, it is difficult, as was shown, to ascertain accurately the amount of alkali salts allowable, but from 40 to 50 grains per United States gallon seems to be the maximum permissible amount in the water to be fed to the boiler. This would allow a concentration of the water until from three to four times the amount

of salts originally present was reached before foaming would be serious from the alkali salts alone, when the water would have to be blown down and replaced with fresh water.

The preceding amount of alkali salt applies to stationary boilers; in locomotive practice, from 30 to 40 grains will soon cause foaming. These amounts are greatly in excess of the soluble salts present in ordinarily softened water, and a water producing these amounts would only be softened in a section having especially hard waters, as certain parts of the West. This removal of the greater portion of the concentrated water and replacement with fresh feed has to be repeated whenever foaming continues for any considerable period. The design of boilers, the degree that the firing is forced, the piping from boilers to engines, and whether the engines are pulling relatively large quantities of steam with reference to boiler capacity, all affect and determine the degree of foaming. This applies especially to stationary plants; locomotive boilers foam more readily than properly designed and operated stationary boilers.

SOLUBILITY OF CERTAIN SUBSTANCES IN WATER

40. The solution of solids causes certain external changes that may be observed readily in many cases. As a solid dissolves in water and assumes the liquid condition, a certain amount of heat is absorbed in the change from the solid to the liquid state, the temperature of the solution falling accordingly. For example, a solution of equal parts of ammonium nitrate and water drops from 40° F. to 4° F., and solutions of various mixtures, mainly of alkaline salts, cause drops in temperature of from 30° F. to 40° F. This drop in temperature, however, is not apparent in dissolving small quantities of salts except when employing special apparatus, etc.

In case the salts or substances dissolved develop chemical action with the solvent, the reverse of the above occurs, and a rise in temperature will result.

41. General Laws in Regard to Solubility.—The following general statements apply to solution, and are sometimes called **laws governing solution:**

1. The quantity of a solid capable of being dissolved in a liquid is strictly limited, and is always the same at the same temperature; or, the coefficient of solubility varies with the temperature.

2. A liquid partly or wholly saturated by one solid is capable of dissolving others that are insoluble or have a less degree of solubility in pure water.

3. The solubility of solids usually increases with the temperature while that of gases decreases. There are many exceptions to the greater solubility of solids with increase of temperature, but hydrogen is the only ordinary gas whose solubility does not decrease with a rise of temperature, it being constant at all temperatures.

42. Salts Used in Water Softening.—Taking up the more important salts met with in water softening, it is found that the carbonates, sulphates, and chlorides of calcium and magnesium comprise the principal ones. A number of other salts are met with and will be considered, but those just mentioned are the most important. It has even been estimated that approximately 95 per cent. of the scale formed in steam boilers is due to the four salts, calcium and magnesium carbonates and calcium and magnesium sulphates. To these salts should be added sodium chloride, as it is of importance where sea-water has to be used and in some localities where salt deposits predominate.

43. Calcium Carbonate.—The salt known as **calcium carbonate,** $CaCO_3$, is not entirely insoluble in water, although it has been so indicated previously. For practical water-softening purposes, however, it may be so considered, as a water containing the maximum amount of the normal carbonate that is soluble is still a soft water. One part of the normal carbonate, $CaCO_3$, dissolves in 28,500 parts of water at ordinary temperatures. This is equivalent to .035 gram per liter, 3.5 parts per hundred thousand, or

2.04 grains per United States gallon. According to Archbutt, only 1.08 grains per gallon remain in solution on removal of all the carbon dioxide present in the water. The remaining carbonate, or soluble amount of calcium carbonate, is not precipitated by caustic lime, showing that the solution is neutral and that it is not held in solution or dissolved by carbon dioxide, the latter precipitating completely with caustic lime. Neither is it thrown down on boiling, as is the calcium bicarbonate.

Water charged with carbon dioxide dissolves calcium carbonate very readily. A solution saturated with carbon dioxide at 0° C. is variously given by different observers as dissolving 1 part of calcium carbonate in from 1,080 to 1,420 parts of water, which is equal to from .704 to .926 gram

TABLE IV

SOLUBILITY OF MAGNESIUM CARBONATE

Pressure Pounds	Parts per Hundred Thousand	Grains per U. S. Gallon
15	621	362.3
30	695	405.0
45	746	435.0
60	904	526.8
75	910	530.2
90	1,316	767.3

per liter, from 70.4 to 92.6 parts per hundred thousand, or from 41.04 to 54.04 grains per United States gallon.

44. Magnesium Carbonate.—The salt called **magnesium carbonate**, $MgCO_3$, goes even more readily into solution in the presence of carbon dioxide than does calcium carbonate, but it is less frequently met with and seldom exceeds 5 or 6 grains per gallon, although up to 30 grains per gallon are occasionally found. While easily removed by caustic lime, magnesium carbonate is much more soluble than is generally supposed, and something like 11 or 12 grains

per gallon may remain in solution unless a further quantity of lime corresponding to that used to throw out the carbon dioxide is added to change the magnesium carbonate to magnesium hydrate.

It has been demonstrated that the solubility of magnesium carbonate depends on the temperature and pressure. The normal carbonate is soluble in water in the absence of carbon dioxide to the extent of from 6 to 12 grains per United States gallon. At 41° F., water saturated with carbon dioxide dissolves magnesium carbonate at various pressures, as shown in Table IV.

The combined effect of temperature and pressure within certain ranges and the effect of temperature, according to Engel and Ville, are given in Table V.

TABLE V

SOLUBILITY OF MAGNESIUM CARBONATE IN WATER SATURATED WITH CARBON DIOXIDE

Pressure Pounds	Temperature Degrees F.	Parts per Hundred Thousand	Grains per U. S. Gallon
15	67	2,780	1,620
31	67	3,310	1,930
47	68	3,730	2,175
69	66	4,350	2,536
82	66	4,620	2,693
91	66	4,850	2,828
110	67	5,120	2,984
132	66	5,660	3,300

A study of Tables IV and V will show, first, the increasing solubility with increase of pressure, and second, the increasing solubility with the elevation of both temperature and pressure.

45. Calcium Sulphate.—Many experiments have been made to determine the solubility of **calcium sulphate,** $CaSO_4$, and the data available show surprising discrep-

ancies. The most exhaustive and authentic experiments in regard to the solubility of calcium sulphate were made by

TABLE VI

SOLUBILITY OF CALCIUM SULPHATE, $CaSO_4 \cdot 2H_2O$

Temperature Degrees F.	Parts per Hundred Thousand	Grains per U. S. Gallon
32	241	140.5
65	259	151.0
75	265	154.0
90	270	157.0
100	272	158.4
106	270	157.6
127	267	156.0
162	256	149.0
187	240	140.0
210	222	129.0

Tilden and Shenstone, jointly, and by Marignac. The results obtained by these investigators are given in Table VI.

The results given in Table VI refer to the hydrated salt, $CaSO_4 \cdot 2H_2O$. The dehydrated salt, $CaSO_4$, is much denser

TABLE VII

SOLUBILITY OF CALCIUM SULPHATE, $CaSO_4 \cdot 2H_2O$ AT HIGH TEMPERATURES AND VARIOUS PRESSURES

Pressure Pounds	Temperature Degrees F.	Parts per Hundred Thousand	Grains per U. S. Gallon
37.8	284.0	78	45.5
80.8	324.5	56	32.7
132.0	356.5	27	15.8
513.5	473.0	18	10.5

and more insoluble. The maximum solidity of the hydrated salt is at about 100° F., with only slight variation in its

solubility between 90° and 127° F. At higher temperature and pressure, Tilden and Shenstone's experiments give valuable data as to the probable behavior of calcium sulphate under boiler conditions. These data are given in Table VII.

The solubility of calcium sulphate is affected by the presence of other salts, especially the chlorides. Calcium chloride decreases the solubility of calcium sulphate, $CaSO_4 \cdot 2H_2O$, while magnesium chloride increases at ordinary temperature, but at higher temperatures has no effect. Sodium chloride and sulphate are generally stated to have no effect on the solubility of calcium sulphate. Carbon dioxide reduces its solubility slightly.

46. Calcium Oxide, or Lime.—The solubility of **calcium oxide,** CaO, or **lime,** deserves consideration owing to its importance as a purifying agent in the shape of caustic lime, $Ca(OH)_2$. Dalton ascertained that lime is far less soluble in hot than in cold water. Practically twice the amount dissolves at 32° F. as at 212° F., contrary to the fact that the solubility of most substances increases with the rise of temperature. The solubility of calcium oxide at various temperatures is given in Table VIII.

TABLE VIII

SOLUBILITY OF CALCIUM OXIDE, *CaO*, AT VARIOUS TEMPERATURES

Temperature Degrees F.	Parts per Hundred Thousand	Grains per U. S. Gallon
60	128.5	75.8
130	100.2	58.4
212	78.7	45.9

A number of conditions affect the solubility of calcium oxide, such as the quality of the lime, the temperature at which it was calcined, the method of preparation, the degree of temperature to which the mass is brought in slaking, and the intimacy of contact of the lime and water in preparing the solution.

In water softening, when caustic lime is used as the lime-water addition, it is customary to take its solubility at 1.25 to 1.3 grams per liter, which is equivalent to 127.5 parts per hundred thousand, or 74.3 grains per United States gallon.

47. Magnesium Sulphate.—The magnesium salt corresponding to the calcium salt, called **magnesium sulphate,** $MgSO_4$, is commonly known as *Epsom salt.* Its solubility is very high, being about 25 per cent. at 32° F., something over 100 per cent. at 212° F., and increasing amounts at higher boiler temperatures and pressures. By itself, magnesium sulphate, mainly from its extreme solubility, is of little importance; but owing to reactions with other salts usually associated together, it is of importance in boiler waters, and this phase of it will be treated later on.

48. Calcium Chloride.—The salt called **calcium chloride,** $CaCl_2$, is highly soluble, dissolving in about one-fourth its weight of water. It is not usually found in natural waters, but is occasionally introduced from artificial sources or in a boiler supply through accident; as for example, where used as a refrigerating brine, it may accidentally reach a feedwater, or where, in manufacturing, it is produced as a by-product.

49. Magnesium Chloride.—Another highly soluble salt, **magnesium chloride,** $MgCl_2$, will dissolve in about three-fourths its weight of cold water, and over $3\frac{1}{2}$ parts, by weight, will dissolve in hot water. Its importance in water purification is due to its decomposition into hydrochloric acid, which produces very serious corrosion.

50. Sodium Chloride.—Common salt, or **sodium chloride,** $NaCl$, is one of the few of the entire list of salts soluble in water whose solubility does not change materially with alterations of temperature. At 60° F., 2.8 parts, by weight, is dissolved; at 140° F., 2.7 parts; and at 220° F., 2.5 parts. It is of importance where sea-water or water from saline wells has to be used to any extent in boilers.

CHEMISTRY OF WATER SOFTENING

51. As described previously, the lime, or Clark, process, the invention of Doctor Clark, is suitable for waters having their hardness due to carbonates of lime or magnesia. All boiler feedwater or water used for any commercial process contains more or less mineral or scale-forming matter in solution. The only exceptions are rainwater and distilled water, and their use is so limited and restricted that they may be ignored entirely. This scale-forming matter is composed mainly of salts of lime, magnesia, iron, alumina, or silica, or of insoluble material, as clay, mud, sand, etc., which, while not scale-forming materials themselves, may, by being held or cemented with the strictly scaling matter, largely increase the bulk of the resulting scale. There are four salts that are of especial importance in scale formation and water softening, namely, *calcium carbonate*, *magnesium carbonate*, *calcium sulphate*, and *magnesium sulphate*. They are the ones most commonly occurring in waters, and it has been estimated that from 95 to 98 per cent. of the boiler incrustations commonly met with are due to the presence of one or all of these in the water used.

52. **Calcium carbonate** is one of the most common compounds in nature, being familiar to all as marble, limestone, chalk, etc. It is perhaps the most common form in which calcium compounds are found in water. It is practically insoluble in pure water, but the presence of carbon dioxide in water rapidly increases its solubility. In nature, the rain washes carbon-dioxide gas from the atmosphere in falling to the earth; then the rainwater coming in contact with carbonates in the soil, or percolating through limestone rocks, takes up calcium carbonate until it is frequently highly charged with this calcium salt. The amount of carbon dioxide in solution may be increased from the soil by acid waters or other reactions decomposing rocks or minerals yielding the gas. It is thus evident in what manner many natural waters have large amounts of carbon dioxide con-

stantly added to them, thereby increasing their solvent action on calcium carbonate.

53. Calcium carbonate does not go into solution strictly as such, but through the agency of the carbon dioxide another salt, namely, *calcium bicarbonate*, is formed. Calcium bicarbonate is expressed by any of the following formulas: $CaCO_3 \cdot CO_2$, $CaO(CO_2)_2$, and $CaH_2(CO_3)_2$. These formulas are identical as to ultimate composition, except that the last one indicates the hypothetical carbonic acid, H_2CO_3, and might be written $CaCO_3H_2CO_3$. They merely express somewhat different conceptions of the formation and constitution of the bicarbonate. To avoid confusion and to fix one formula in the mind, the first formula, $CaCO_3 \cdot CO_2$, will be used in all equations showing reactions, as it perhaps expresses more clearly and simply the composition of the bicarbonate. This second portion of carbon dioxide is not so securely a part of the bicarbonate as is the CO_2 of the normal carbonate. The former is a less stable salt than the latter, and the CO_2 in the compound $CaCO_3 \cdot CO_2$, is loosely held; "half bound" is a common expression, and well describes its condition. It may be removed by boiling or by the addition of many hydrates, such as calcium hydrate, $Ca(OH)_2$; sodium hydrate, $NaOH$; potassium hydrate, KOH; barium hydrate, $Ba(OH)_2$; etc. As the calcium carbonate is held in solution by the additional CO_2, the removal of this renders the calcium carbonate insoluble; hence, boiling or removal by precipitation removes from solution all lime except the small amount soluble in pure water. Owing to its low cost, caustic lime, $Ca(OH)_2$, is the reagent almost universally used to remove lime held as bicarbonate.

54. Magnesium carbonate, $MgCO_3$, is much like the corresponding calcium compound in its general relations of solubility and removal. It is more soluble than calcium carbonate, but is seldom found above 5 or 6 grains per gallon in any other water except mineral waters. It enters ordinary waters principally from the solution of dolomite, the double carbonate of calcium and magnesium corresponding to lime-

stone. These two carbonates usually occur together, and their removal is effected in the same manner by caustic lime. At high temperatures, magnesium carbonate is decomposed into the hydrate, $Mg(OH)_2$, and carbon dioxide. The hydrate is much more insoluble than the carbonate; hence, the course is suggested of converting the latter into the former. This is readily accomplished by adding as much more lime as is required to combine with the carbon dioxide, CO_2, which forms the hydrate according to the following reactions:

$$MgCO_3CO_2 + Ca(OH)_2 = CaCO_3 + MgCO_3 + H_2O$$
$$MgCO_3 + Ca(OH)_2 = CaCO_3 + Mg(OH)_2$$

Combining the reactions into one equation, the following equation is obtained:

$$\underset{\text{(soluble)}}{MgCO_3CO_2} + 2\underset{\text{(soluble)}}{Ca(OH)_2} = 2\underset{\text{(insoluble)}}{CaCO_3} + \underset{\text{(insoluble)}}{Mg(OH)_2} + H_2O$$

55. Calcium sulphate, $CaSO_4$, is generally present to some extent in most waters. The most common form in which it occurs in nature is gypsum, or plaster of Paris. It is readily soluble up to about 150 grains per gallon at ordinary temperatures. Calcium sulphate is not thrown out of solution until the water is heated considerably above the boiling point, say to about 55 pounds boiler pressure, or 300° F., when it precipitates in the form of heavy crystals, producing an extremely hard, cement-like deposit, the most objectionable of all boiler scales. As the sulphate deposits very slowly, this is an important reason for its adhering so firmly to plates and tubes. The salt being rather readily soluble in water, the use of a softened water will soon begin to soften sulphate scale previously deposited in boilers, and if an excess of alkali is present, it also will assist in softening the scale.

56. Magnesium sulphate, $MgSO_4$, is the salt of magnesium corresponding to calcium sulphate, its most familiar form, as previously stated, being Epsom salt. It is very soluble in water, and does not of itself form scale. However, under boiler conditions and in the presence of calcium carbonate, these compounds react together to form calcium

sulphate and magnesium carbonate, as shown in the following equation:

$$\underset{\text{(soluble)}}{CaCO_3,CO_2} + \underset{\text{(soluble)}}{MgSO_4} = \underset{\text{(insoluble)}}{CaSO_4} + \underset{\text{(insoluble—partly)}}{MgCO_3,CO_2}$$

The magnesium carbonate formed in the presence of caustic lime or other hydrate reacts as previously shown. It is interesting to note that magnesium sulphate is generally believed to be formed in nature by the action of calcium-sulphate water on magnesium carbonate, or just the reverse of the preceding reaction.

57. Miscellaneous Salt Occurring in Water.— In addition to the salts just described, a number of other salts and free acids occur in some waters used for industrial purposes. The most important of the former are *calcium* and *magnesium chlorides*, both of which are corrosive. Chloride of magnesium particularly decomposes at relatively low temperatures into hydrochloric acid and magnesia, as shown by the following equation:

$$MgCl_2 + H_2O = 2HCl + MgO$$

Calcium chloride is more stable than magnesium chloride, but the high temperatures and pressures of steam boilers are believed to decompose it also, forming hydrochloric acid, which, of course, is extremely corrosive. Both magnesium sulphate and carbonate may react with alkaline chlorides and form free acid, but such reactions are seldom met with in water-softening practice. Nitrates, as well as a few other salts, are sometimes present in such waters as are being considered, but their presence is either so limited in amount or so rare an occurrence that only reference will be made to them.

58. Free Acids Occurring in Water. — The free acids are present originally as such in the water, or they are caused by the decomposition of their salts under boiler conditions. Similar to the decomposition of calcium and magnesium chlorides, certain nitrates and sulphates may be broken up. Sulphuric acid and ferrous sulphate are the chief corrosive compounds in the latter class. They result

from the oxidation of pyrites from coal mines, and in many localities they are caused by the large amounts of waste liquors from washing and pickling operations, the latter principally from cleaning scale from iron and steel. In all coal-mining sections, the streams are more or less contaminated by the ferrous sulphate and free sulphuric acid formed from the pyrites. Not infrequently, small streams with large mining operations draining into them are so foul that the waters from them are unfit for use in boilers even with treatment, as the soluble salts in the latter case would be too high. Owing to the pollutions just mentioned, the water from a number of large rivers in the coal-mining regions requires treatment before it is a suitable boiler supply.

REAGENTS AND COAGULANTS

CHEMICAL REAGENTS

59. By far the larger part of all commercial water-softening today is done by means of only *lime* and *soda ash*, as these are the cheapest materials available and give the greatest efficiency per unit of cost.

60. Lime.—The reagent first used in commercial softening, and in some respects the most important, is **caustic lime**, $Ca(OH)_2$. This reagent is applied as slaked lime, either as the milk or cream of lime (having a large excess of the hydrate in suspension), or as a saturated lime water. Either method gives satisfactory results, and each has certain points of superiority. As previously explained, caustic lime precipitates the bicarbonates of lime and magnesia according to the following reaction:

$$\underset{\text{(soluble)}}{CaCO_3CO_2} + \underset{\text{(soluble)}}{Ca(OH)_2} = \underset{\text{(insoluble)}}{2CaCO_3} + H_2O$$

One hundred parts, by weight, of the normal carbonate, $CaCO_3$, requires 56 parts, by weight, of CaO. As a convenience in calculation, the $CaCO_3$ is taken instead, and, for the same reason, CaO is taken instead of the caustic lime; the

same course is followed with $MgCO_3$, the caustic taking up the "half-bound" CO_2 and forming the insoluble normal carbonate. In the case of carbonate of magnesia, two equivalents, or molecules, of lime is required to convert the more soluble carbonate into the insoluble hydrate; thus,

$$\underset{\text{(soluble)}}{MgCO_3.CO_2} + \underset{\text{(soluble)}}{2Ca(OH)_2} = \underset{\text{(insoluble)}}{2CaCO_3} + \underset{\text{(insoluble)}}{Mg(OH)_2} + H_2O$$

Eighty-four parts, by weight, of $MgCO_3$ requires 112 parts, by weight, of lime (CaO).

Lime purification is the cheapest known, and for carbonate waters to which it is adapted or waters having all their hardness temporary, nothing is more efficient. It may also be used to neutralize free acids or ferrous sulphate or chloride, or magnesium chloride. But, in these cases, calcium sulphate is formed and produces a scale formation that may be serious, or the resulting salt will be decomposed and produce corrosion. A slight excess of lime over the theoretical quantity required assists in the rapid settling of the precipitate and gives a causticity that insures the completion of the lime reaction. Any considerable excess must be guarded against, as it may be more troublesome than the carbonate originally in solution.

61. Sodium Carbonate.—The water-softening material called **sodium carbonate,** Na_2CO_3, or **soda ash,** is used mainly with the idea of breaking up chlorides and sulphates and of neutralizing free acids. The equation that represents the action of sodium carbonate on calcium sulphate is as follows:

$$\underset{\text{(soluble)}}{CaSO_4} + \underset{\text{(insoluble)}}{Na_2CO_3} = \underset{\text{(insoluble)}}{CaCO_3} + \underset{\text{(soluble)}}{Na_2SO_4}$$

From this equation it will be seen that 136 parts, by weight, of calcium sulphate requires 106 parts, by weight, of sodium carbonate to make the reaction complete. The reaction with magnesium sulphate corresponds exactly to the equation given for calcium sulphate, 106 parts, by weight, of sodium carbonate being required for every 120 parts, by weight, of magnesium sulphate.

In the presence of free sulphuric acid or ferrous sulphate, the reactions that take place may be expressed by the following equations:

$$H_2SO_4 + Na_2CO_3 = H_2CO_3 + Na_2SO_4$$
$$FeSO_4 + Na_2CO_3 = FeCO_3 + Na_2SO_4$$

From these it will be seen that 96 parts, by weight, of sulphuric acid, or 152 parts, by weight, of ferrous sulphate, requires 106 parts, by weight, of sodium carbonate.

The reaction between H_2SO_4 and Na_2SO_4 includes no insoluble compounds and produces none. The theoretical compound H_2CO_3 is decomposed at once into CO_2 and H_2O.

All compounds entering into the reaction between ferrous sulphate and sodium carbonate are soluble, as are the resulting compounds, the $FeCO_3$ being rapidly changed into iron oxide, FeO, and CO_2.

The reactions with chloride and nitrate of calcium may be expressed by the following equations:

$$CaCl_2 + Na_2CO_3 = CaCO_3 + 2NaCl$$
$$\underset{\text{(soluble)}}{Ca(NO_3)_2} + \underset{\text{(soluble)}}{Na_2CO_3} = \underset{\text{(insoluble)}}{CaCO_3} + \underset{\text{(soluble)}}{2NaNO_3}$$

The corresponding magnesium salts give analogous reactions. Sodium carbonate also reacts with calcium or magnesium carbonate, forming soluble sodium bicarbonate and insoluble calcium or magnesium carbonate, according to the following equation:

$$CaCO_3CO_2 + Na_2CO_3 + H_2O = CaCO_3 + 2NaHCO_3$$

The sodium bicarbonate thus formed is decomposed by the heat as follows:

$$2NaHCO_3 = Na_2CO_3 + CO_2 + H_2O$$

62. Most waters will require both lime and soda ash, and these two reagents are generally used together. There are few waters that will not respond to one or the other of these reagents or their combined action. The lime removes the carbonates of lime and magnesia held as bicarbonates. It also reacts with magnesium sulphate, chloride, and nitrate, and free acids and ferrous sulphate; but as the same quantity

of soda ash is required to break up the resulting salt as if lime had not been used [the magnesium salts all require a molecule of lime in addition to the soda ash, to form the insoluble $Mg(OH)_2$], soda ash is used as just stated. So that there may be no chance of misunderstanding, it will be well to repeat that these two reagents meet practically every case of commercial water softening. The only exception is where the soluble salts not removable, either present or produced by the reactions, are excessive, when reagents yielding insoluble salts have to be used.

63. Caustic Soda.—As a water softener, **caustic soda,** $NaOH$, or **sodium hydrate,** has had quite an extended use in the past, but owing to its comparatively high price, it is seldom employed at present. It reacts with almost all scale-producing salts, as well as with free acids. The reactions correspond with those given for caustic lime, $Ca(OH)_2$, and for the carbonates, sulphates, etc., except that two equivalents, or molecules, of $NaOH$ have to be substituted for every equivalent, or molecule, of $Ca(OH)_2$.

If otherwise desirable, caustic soda could not be used alone for a carbonate water, owing to the fact that sodium carbonate would be formed and left in solution, making the water too alkaline. But if calcium sulphate is present, the sodium carbonate will be available for further reaction as if added. Neither is caustic soda available for the removal of calcium sulphate alone, as caustic lime, $Ca(OH)_2$, is formed, and unless bicarbonates are present to take up this caustic lime, too much of this compound remains dissolved in the water. If used to precipitate calcium and magnesium carbonates, sodium carbonate is formed, and this has to be taken account of in softening. Soda ash is extremely caustic, is inconvenient to handle, and takes up moisture and carbon dioxide very readily.

When lime is slaked and mixed with soda ash, preparatory to adding the two together to water, some of the soda is causticized according to the following reaction:

$$Ca(OH)_2 + Na_2CO_3 = 2NaOH + CaCO_3$$

This, however, is not objectionable, as no efficiency is lost and the calculations for the addition of the reagents are not affected.

64. Barium Salts.—In many respects, **barium compounds** are the ideal water-softening reagents, owing to the fact that no soluble salts resulting from the reactions are left in the water. The only drawback to their extensive use is the cost and their high molecular weights, requiring a large amount for a given precipitation. Their use is therefore restricted to waters that carry such a mass of alkaline salts that an increase would cause foaming quickly; or to water the scale-forming salts of which are so high that the softening reactions, using lime or soda salts, produce a prohibitive amount of soluble salts. The principal salts in this class are *barium hydrate*, $Ba(OH)_2 + 8H_2O$, and *barium carbonate*, $BaCO_3$. The chloride and aluminate have been used in special cases, but they are of no importance. The aluminate breaks up both carbonates and sulphates, all resulting salts being insoluble.

65. A few years ago claims were made that **barium hydrates** would supplant most softeners, and had the assumptions as to cost been made good, the claims would no doubt have been largely realized. The reactions of this compound correspond to those of caustic lime and caustic soda. Practically all scale-forming and corroding salts and acids are broken up by it.

The reactions that take place between various salts and acids in the water may be seen by the following equations:

$$\underset{\text{(soluble)}}{CaCO_3{\cdot}CO_2} + Ba(OH)_2 = \underset{\text{(insoluble)}}{BaCO_3} + \underset{\text{(insoluble)}}{CaCO_3} + H_2O$$

$$\underset{\text{(soluble)}}{MgCO_3{\cdot}CO_2} + Ba(OH)_2 = \underset{\text{(insoluble)}}{BaCO_3} + \underset{\text{(insoluble)}}{MgCO_3} + H_2O$$

$$\underset{\text{(insoluble)}}{CaSO_4} + Ba(OH)_2 = \underset{\text{(insoluble)}}{BaSO_4} + \underset{\text{(soluble)}}{Ca(OH)_2}$$

$$\underset{\text{(insoluble)}}{MgSO_4} + Ba(OH)_2 = \underset{\text{(insoluble)}}{BaSO_4} + \underset{\text{(soluble)}}{Mg(OH)_2}$$

$$CaCl_2 + Ba(OH)_2 = BaCl_2 + Ca(OH)_2$$
$$MgCl_2 + Ba(OH)_2 = BaCl_2 + Mg(OH)_2$$
$$2HCl + Ba(OH)_2 = BaCl_2 + 2H_2O$$
$$FeSO_4 + Ba(OH)_2 = BaSO_4 + Fe(OH)_2$$
$$H_2SO_4 + Ba(OH)_2 = BaSO_4 + 2H_2O$$

Barium hydrate is not only expensive, but more of it is required for removing a given quantity of scaling. From the preceding equations it will be seen that 171 parts, by weight, of $Ba(OH)_2$ is required to remove 100 parts of $CaCO_3$ or 84 parts of $MgCO_3$, while 66 and 112 parts of CaO perform the same service. It requires 106 parts of soda ash to remove 152 and 98 parts, respectively, of ferrous sulphate and sulphuric acid, while 171 parts of barium hydrate is required.

66. Barium carbonate is used to some extent with waters high in acid or ferrous sulphate, reactions corresponding to those given for barium hydrate occurring. It may be used with other sulphates, but since it is so nearly insoluble, the reactions are very slow and an excess has to be thrown into the reaction tanks. This, however, is harmless, as no more is dissolved than that which enters into reaction with the SO_4 of the acid or salt.

67. Trisodium Phosphate.—As distinguished from reagents used outside the boiler, **trisodium phosphate,** $Na_3PO_4 \cdot 12H_2O$, is a boiler compound. It can be used efficiently only at a boiling temperature, and is therefore not used outside the boiler or heater. All lime and magnesia salts are precipitated under boiler conditions as a light, bulky sludge of phosphates. The reactions correspond to the one here given for calcium sulphate:

$$3CaSO_4 + 2Na_3PO_4 = 3Na_2SO_4 + Ca_3(PO_4)_2$$

It is claimed that much less than the theoretical quantity will convert all lime and magnesia salts to a non-adherent sludge that is easy to blow out. While this is questionable, sodium phosphate is one of the best and most efficient boiler compounds available.

68. Sodium Fluoride.—The use of **sodium fluoride**, *NaF*, was patented by the late Dr. C. A. Doremus, the well-known chemist. It is only adapted for use in the boiler, as the fluorides are not precipitated in cold water. The claim is also made for this material that much less than the theoretical amount is thoroughly efficient, and that one-fourth of this amount will give a fluoride sludge that develops no tendency to stick to tubes or sheets and blows out readily. Where the steam must be free from all impurities, it has advantages over any other compound, as nothing volatile passes over with the steam. Its price, however, is prohibitive, so that it has no place in ordinary commercial softening. But it may be used in certain special cases, and is of interest from a technical standpoint.

69. Miscellaneous Salts Used as Reagents.—While a number of other salts have been used as reagents for water softening, none of them has reached any technical or commercial importance. Among these are alkali oxalates, chromates, and sodium silicate, their very limited use being entirely discontinued, either owing to high cost or to imperfect results, or to both. *Magnesium*, as the hydrate, $Mg(OH)_2$, has had considerable use, mainly in Germany, but is not known to have been used in America. The reactions correspond to those of caustic lime for bicarbonates, and take place only in hot water, and the magnesium carbonate formed reacts with calcium sulphate; thus,

$$MgCO_3 + CaSO_4 = CaCO_3 + MgSO_4$$

Thus, instead of sodium sulphate, as in the soda-ash reaction, magnesium sulphate is left in solution. Technically, the use of magnesium does not appear to be so advantageous as lime and soda ash and, besides, it is costlier.

COAGULANTS

70. Purpose of Coagulants.—Frequently, if sedimentation alone is relied on for clarification, the treated water will not filter clear, or the precipitate will not settle out.

This is quite as often due to finely divided silt carried by the water as to a precipitate from softening. The same difficulty occurs in filtering untreated supplies and is corrected in the same way, namely, by the addition of a *coagulant*. The efficiency of a coagulant depends on the gelatinous precipitate formed, which carries down with it the matters in suspension.

71. Coagulants Used.—The coagulants are **ferrous sulphate,** $FeSO_4$, and **aluminum sulphate,** $Al_2(SO_4)_3$. In order to be effective, there must be either sufficient hydrates or carbonates present in the water or added with the coagulant to decompose the salts according to the following equations:

$$FeSO_4 + Ca(OH)_2 = Fe(OH)_2 + CaSO_4$$

$$FeSO_4 + Na_2CO_3 + H_2O = Fe(OH)_2 + Na_2SO_4 + CO_2$$

$$Al_2(SO_4)_3 + 3Ca(OH)_2 = Al_2(OH)_6 + 3CaSO_4$$

$$Al_2(SO_4)_3 + 3Na_2CO_3 + 6H_2O = Al_2(OH)_6 + 3Na_2SO_4 + 3CO_2$$

Any of the alkaline or alkaline-earth hydrates or carbonates react in a similar manner. In case an addition has to be made, as is usually the case, soda ash, Na_2CO_3, is generally used when water is being softened, as the alkaline-earth salts produce calcium sulphate requiring the same amount of soda ash as the ferrous sulphate. It is essential that the necessary hydrate or carbonate be present, or sulphuric acid will result from the decomposition of either iron or aluminum sulphate. In softening practice, the amount of coagulant varies with the condition of the water, but an approximate amount is 1 grain per gallon, or about .75 pound per 4,000 gallons; this will require approximately $\frac{1}{2}$ pound of soda ash or $\frac{1}{4}$ pound of lime. If the water contains any considerable amount of calcium-carbonate sludge, this acts as a very efficient coagulant, and the iron or alum salt will seldom be required.

72. In the filtration of municipal supplies, coagulants are more commonly used than in water softening. Caustic lime is usually added to form the hydrate, as calcium sulphate thus added to the water is seldom of consequence or is as objectionable as sodium sulphate; also, the soda ash

required would add an appreciable item of expense to the filtration of a large city supply. An even more important reason for a coagulant in the latter case is that it forms a film on the surface of the sand filters and entraps the bacteria. In many cases, over 99 per cent. of the bacteria is removed. Even in settling basins or tanks, the sludge removes a very large percentage of the bacteria. In waters colored by organic impurities collected from peaty watersheds, etc., the coagulant removes the coloring matter and gives a clear, colorless product.

TABLE IX

QUANTITY OF REAGENTS REQUIRED TO PRECIPITATE SALTS

To Precipitate 1 Part of	Amount of Reagents Required						
	CaO 100 Per Cent.	Na_2CO_3 58 Per Cent. Na_2O	$NaOH$ 74 Per Cent. Na_2O	Na_3PO_4 12 H_2O 100 Per Cent.	$Ba(OH)_2$ 56 Per Cent.	$BaCO_3$ 100 Per Cent.	NaF 100 Per Cent.
$CaCO_3$ as bicarbonate	.560	1.060	.837	2.530	3.060		.840
$MgCO_3$ as bicarbonate	.660	1.262	.997	3.015	3.642		1.000
$CaSO_4$		.786	.616	1.870	2.250	1.449	.618
$MgSO_4$		.890	.698	2.111	2.555	1.642	.700
$CaCl_2$		.962	.754	2.283	2.756		.756
$MgCl_2$		1.124	.881	2.666	3.221		.884
CO_2	1.272	2.410	1.818	5.757	6.727		
H_2SO_4	.571	1.080	.816	2.583	3.121	2.010	
$FeSO_4$	.368	.693	.519	1.666	2.000	1.290	.553

73. The quantity of the various reagents required to precipitate the usual salts and acids is given in Table IX, and the relative costs of these reagents in Table X.

74. Tables IX and X show that lime and soda ash are commercially superior to any other reagents, as they will soften with equal efficiency practically any water that should be treated. In regard to sodium phosphate, sodium fluoride, and barium hydrate, it should be stated that they are only used as boiler compounds, and that the use of less than the theoretical amounts prevents the formation of scale; thus,

the comparison is less unfavorable than shown. All calculations in Tables IX and X are based on molecular quantities and the completion of the reaction involved.

75. As shown in Table IX, lime used as $Ca(OH)_2$ precipitates $CaCO_3$, $MgCO_3$, $FeSO_4$, and CO_2, and neutralizes free acids. It also reacts with other magnesium salts, but in such cases it leaves a lime salt to be broken up by soda ash. It should be as pure and fresh as possible. A "fat" lime, that is, one high in CaO and having the smallest amount of

TABLE X

COST OF REAGENTS REQUIRED TO PRECIPITATE SALTS

To Precipitate 1 Pound (7,000 Grains) of	CaO per Pound 3c. Cent	Na_2CO_3 per Pound 1c. Cents	$NaOH$ per Pound 2.6c. Cents	$Na_3PO_4 12H_2O$ per Pound 3.25c. Cents	$Ba(OH)_2$ per Pound 2.5c. Cents	$BaCO_3$ per Pound 1c. Cents	NaF per Pound 5c. Cents
$CaCO_3$ as bicarbonate	.168	1.060	2.176	8.222	7.650		4.20
$MgCO_3$ as bicarbonate	.198	1.262	2.592	9.799	9.105		5.00
$CaSO_4$		.786	1.602	6.077	5.625	1.45	3.09
$MgSO_4$		.890	1.815	6.861	6.387	1.64	3.50
$CaCl_2$		.962	1.960	7.420	6.890		3.78
$MgCl_2$		1.124	2.291	8.665	8.052		4.42
CO_2	.382	2.410	4.727	18.710	16.817		
H_2SO_4	.171	1.080	2.122	8.395	7.802	2.01	
$FeSO$	.110	.693	1.349	5.414	5.000	1.29	2.76

impurities, is desirable. As a rule, 90 per cent. of CaO is all that can be counted on for lime of good quality, and frequently the amount is less than this. Tables IX and X assume that 100 per cent. of CaO is present, and the necessary correction must be made for whatever impurities are contained in the lime.

To determine the CaO, weigh out a .28-gram powder of a properly selected sample and add it to 100 cubic centimeters of a 10-per-cent. cane-sugar solution in a flask; then shake until no further solution takes place. Filter off 20 cubic centimeters and titrate with $\frac{N}{50}$ sulphuric acid, using phenol phthalein as an indicator. The number of cubic

centimeters used is the per cent. of CaO. Any other standard strength of acid may be used and the calculations made accordingly.

76. Soda ash breaks up the sulphates, chlorides, nitrates, and free acids present, including CO_2. It is sold in the trade as a 58-per-cent. ash, which means 58 per cent. of Na_2O, or about 98 per cent. of Na_2CO_3. A purity of 95 per cent. is generally taken for water-softening calculations. Tables IX and X assume 100 per cent. To determine strength of the ash, dissolve 1.06 grams of a properly taken average sample in water, dilute to any convenient exact bulk, and take one-tenth accurately. Then titrate this (.106 gram) with $\frac{N}{50}$ sulphuric acid, using methyl orange as an indicator. The number of cubic centimeters used is the per cent. of Na_2CO_3.

77. Caustic soda reacts with all the compounds that are ordinarily removable and is one of the best reagents, but its cost is about double that of soda ash for the same degree of purification. It is sold in drums, usually of 74 per cent. Na_2O, and its strength is determined in the same way as that of soda ash.

The other reagents are generally of the strength shown and can be tested by regular analytical methods; their use, however, is not general enough to necessitate giving the tests here. The prices given in Table X are p[illegible] average prices, but of course they fluctuate and var[illegible] different sections of the country.

78. Calculations of Reagents and Control of Softening.—From the data given, the calculation of softening additions is quite simple, having obtained, of course, a correct analysis of the water. In American water-softening practice it is more general to express constituents in grains per United States gallon, though parts per hundred thousand is much used. Parts per million is very little used, except in sanitary analysis. Grains per United States gallon are changed to parts per hundred thousand by dividing by .583 or multiplying by 1.715. Thus, referring to Table IX, take

the amount given opposite each constituent of the water under the reagent it is desired to find the quantity required, and multiply this factor by the number of grains per gallon; this gives grains of the reagent required per gallon, which multiplied by 1,000 and divided by 7,000 (grains per pound), gives pounds per 1,000 gallons. For convenience, dividing the grains required per gallon by 7 gives pounds per 1,000 gallons. As pounds per 1,000 gallons is the most expeditious form to get at reagents required, Table XI will be found convenient. To use this table, simply multiply the factors given under each reagent and opposite the desired constituent by the grains per gallon as shown by the analysis.

TABLE XI

DATA FOR DETERMINING THE QUANTITY OF REAGENT REQUIRED TO PURIFY WATER

Salts to be Removed	Reagents						
	CaO	*Na_2CO_3*	*NaOH*	*Na_3PO_4*	*$Ba(OH)_2$*	*$BaCO_3$*	*NaF*
$CaCO_3CO_2$	.080	.151	.120	.361	.437		.120
$MgCO_3CO_2$	.094	.180	.142	.431	.520		.143
$CaSO_4$		.112	.088	.267	.321	.207	.088
$MgSO_4$		.127	.100	.301	.365	.235	.100
$CaCL_2$		.137	.108	.326	.394		.108
$MgCL_2$		.161	.126	.381	.460		.126
CO_2	.182	.344	.260	.822	.961		
H_2SO_4	.082	.154	.117	.369	.446	.287	
$FeSO_4$	.053	.099	.074	.238	.286	.184	.079

WATER SOFTENING
(PART 2)

WATER-SOFTENING APPARATUS

INTERMITTENT AND CONTINUOUS APPARATUS

1. As was stated in *Water Softening*, Part 1, two processes for the purification, or softening, of water have been in use for many years. The *intermittent process* dates back to 1841, and began with Clark's water-softening investigation, while the *continuous process* was not invented until about 1866 or 1867. The development of the continuous process into its present state of efficiency, as well as the development of the various forms of apparatus used in this process, has been gradual and is the result of the work of many chemists and engineers. Today both processes are in very general use, and it can hardly be claimed that one supplants the other.

It should be clearly understood that either method, provided the apparatus is properly designed and constructed and the treatment is carefully carried out, is capable of producing water that will be satisfactory for any purpose for which soft water is required. The selection of a water-softening process, however, depends mainly on local conditions, and each installation demands careful study in order to determine which type of apparatus is the most suitable.

2. Advantages of the Intermittent Type of Apparatus.—In the intermittent type of apparatus, each tankful of water is treated separately. In this way, more perfect

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control of softening is possible, as there is always a definite volume of water to which may be added exact amounts of reagents. The amounts of reagent are constant or variable, as the character of the water to be treated is regular or changes from time to time. The ease with which reagents could be varied was formerly a very strong point in favor of the intermittent type of apparatus, but with the development and simplification of apparatus for accurately proportioning additions of reagents to the flow of raw water, it cannot be held as a serious objection to the continuous type, although it is still met with from advocates of the intermittent system. Another advantage claimed, namely, that part of the precipitated matter may be removed by subsidence and the remainder by filtration better than in the continuous type, is probably the principal one of the intermittent over the continuous system, and under certain conditions it becomes an important factor to be considered.

3. Advantages of the Continuous Type of Apparatus.—Offsetting the points in favor of an intermittent apparatus, the continuous type of apparatus does not require the large capacity in settling and filtering tanks, and, besides, it furnishes a supply of softened water continuously and regularly. Also, in large installations, the apparatus requires less attention, as there are no corresponding valves to open and close, the water coming over continuously and regularly to the storage tanks.

4. Points to Be Considered in Installing Apparatus.—As has been stated, either the intermittent or the continuous type of apparatus is suitable for treating any water that can be softened profitably. In recognition of this fact, it may be stated that most of the manufacturers of water-softening apparatus now make both types of apparatus, so as to be prepared to furnish the one better adapted to local conditions or to meet the design demanded.

The essential points to decide the installation of one or the other systems of water softening would seem to be, briefly, the space available and the cost of installation. The

former is altogether a local condition to be decided in each individual case, while the latter to a certain extent, and more especially in large installations, depends on the cost of materials available—whether wood, steel, or concrete—for constructing the treating and storage tanks. The cost of operation is not essentially different for the two systems. In small plants, where the necessary attention can be given by the pumpman, the water or boiler tender, or some other regular employe, the intermittent apparatus may require a little less time, but the difference is unimportant. In plants of larger size, that is, those requiring the attendant's entire time for the softening operation, the two types of apparatus are about balanced. In very large installations, the continuous type has the advantage of requiring less attendance, as one man can take care of a continuous softener of the largest type, while an intermittent apparatus handling the water for a large municipal or manufacturing plant of very large boiler capacity may require an additional attendant per turn. This, however, may be offset by the maintenance of the appliances in connection with the continuous softener, for while they are for the most part simple and positive in action, yet, as in all mechanical appliances, they will wear out and require repair or adjustment.

DESCRIPTION OF INTERMITTENT APPARATUS

5. Simplest Form of Intermittent Apparatus.—The simplest form of intermittent water-softening apparatus is shown in plan and elevation in Fig. 1. It consists of two or more plain tanks *A* and *B* of the desired capacity, on top of which is located a simple solution tank *C* for dissolving the lime and soda ash. Each tank is provided with a stirring apparatus. The stirrer of the solution tank, being small, is operated by hand, while that of the large tanks is run by power. The stirring arrangement of the large tanks consists of a vertical shaft *a* and a horizontal shaft *b*, with bevel gears *c* and *d* and two or more blades or paddles *e* at the bottom reaching nearly to the circumference of the tanks.

Power to drive the shafts is applied through belting to the pulley *f*. In the tanks shown, no filters are used, sedimentation alone being relied on to remove the sludge.

Floating discharge pipes *g* are frequently used for withdrawing the water to the boiler feed-lines from the surface

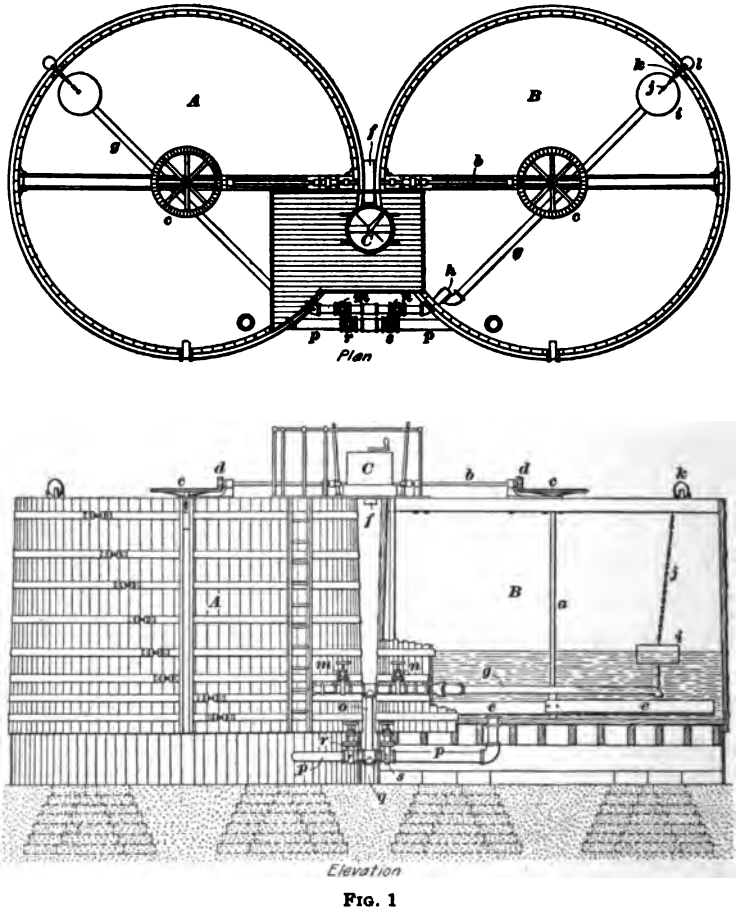


FIG. 1

of the water. This is done to give the precipitated matter a longer time to settle. These discharge pipes have a swivel joint in the fitting *h*, and their free end is connected by a chain to a hollow float *i*. Obviously, the free end will rise

and fall with the water level. A chain *j* is attached to the top of the float *i* and passes over the pulley *k* and to the outside of the tank. To the end of the chain is attached a small weight, which, by its position, indicates the water level in the tank.

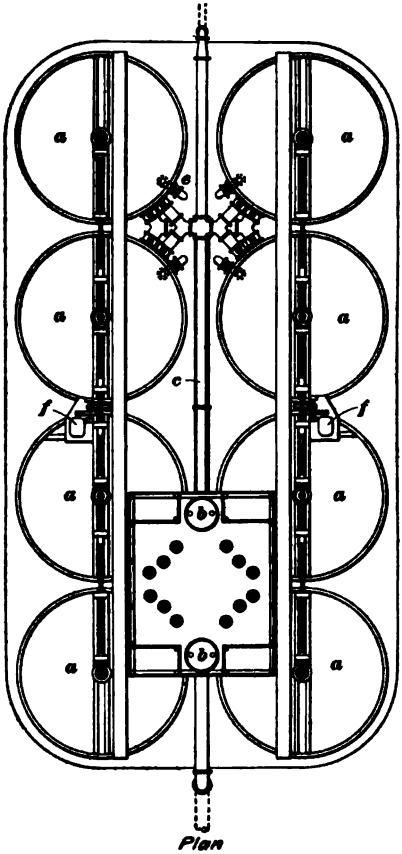
The different tanks are usually so arranged that either one can be used at will. Thus, by opening valve *m* and closing valve *n*, the purified water can pass from the tank *A* to its destination.

The tanks can be emptied and the sludge removed through the pipes *p*, which lead to a single blow-off *g*. Each pipe *p* has its own valve *r* and *s*, respectively, so that either tank can be emptied independently of the other.

6. Air Agitation.—In Fig. 1, the agitation of the water and reagents in the settling tanks is accomplished by rotating blades or paddles, as shown. Agitation, however, may also be accomplished by means of compressed air. For this purpose, a perforated pipe enters the tank at its bottom. In use, the air blast is turned on either when adding the reagents or after they are added; it is continued for a sufficient period to agitate the contents of the tank thoroughly and to mix the precipitants with the water. With an air pressure of 8 ounces per foot depth of water, this mixing is, as a rule, accomplished in from 15 to 30 minutes.

7. Economical Advantages of Air Agitation.—At most plants, even small ones, air pressure is available either from some form of blower or from a pressure system. If no compressed air is available, a small and relatively inexpensive blower or compressor may be installed to furnish the necessary air. Such a device can be installed and operated at less expense than a mechanical stirring device can be installed and operated.

Air agitation is the most convenient method to use in mixing the chemicals and aiding the settling of the precipitated matter. Objections based on the fact that a certain amount of carbon dioxide is introduced from the air have been made, but they may be ignored, as actual experience



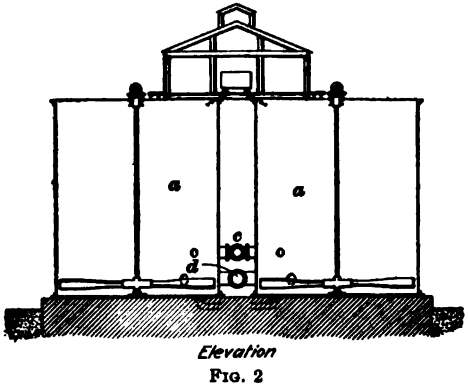


FIG. 2

has proved that this comparatively small amount of carbon dioxide and its consequent reactions with the compounds formed by the impurities of the water with the reagents does not interfere with the efficient and economical working of this method.

8. Eight-Purification-Tank System.—In Fig. 2 is shown an arrangement of an intermittent purification plant having eight precipitating, or purification, tanks *a*, and two or more solution tanks *b*. This arrangement permits the use of two reagents separately. Each solution tank is connected to each of the precipitation tanks by means of pipes, not shown, and each of the pipes has a valve for controlling the solution. The purified water is conveyed to its destination by the pipe *c*, which has branches leading to each tank. Each branch has its own valve. The raw water enters through the pipe *d*, which also has branches, with valves therein, leading to each tank. Each tank can be emptied through a valved pipe *e* that enters the tanks at the bottom. The agitation in the precipitation tanks is accomplished by means of paddles driven by shafting, as described in connection with Fig. 1. In this case, however, the shafting is driven directly by electric motors *f*, geared to the horizontal shafts.

Owing to the manner in which the tanks are connected to the pipes *c* and *d*, the plant can be run with any one tank, several tanks, or all tanks at the same time.

9. Reagents Used in the Eight-Tank Apparatus. In the eight-tank apparatus, one of the solution tanks carries milk of lime, or cream of lime, and the other a solution of soda ash. These reagents are prepared in the tanks *b* and are admitted at will to the precipitating tanks *a*.

It will be observed that in the apparatus shown in Fig. 2 milk of lime is used, while in the apparatus shown in Fig. 1, lime water is employed. The difference between these reagents is that lime water is simply a saturated solution of lime in water, while milk, or cream, of lime is a supersaturated solution of lime in water, or, in other words,

a saturated solution of lime water carrying an excess of lime in suspension. The action of milk, or cream, of lime is the same as that of lime water, as, through dilution, the former becomes the latter when admitted to the large precipitation tanks.

Lime water, however, is frequently used in the type of apparatus shown in Fig. 2. In fact, it is claimed by many that a more regular addition of caustic lime and better control of the lime are secured by the use of lime water than by the use of cream of lime. There is no good reason why accurate control cannot be maintained by using either lime water or milk of lime.

10. Capacity of Eight-Tank Apparatus.—The tanks *a*, Fig. 2, have a capacity of 150,000 gallons each, and, ordinarily, they can be emptied four times in 24 hours. The entire capacity of each tank, however, is not available, as the sludge must be left on the bottom. To keep the sludge on the bottom and thus avoid discharging it into the feedwater, about 15 to 20 per cent. of the water is left in each tank, giving in the case cited, an available capacity of 125,000 gallons per tank. If emptied four times each 24 hours, the capacity of each tank is equal to 500,000 gallons, and the total capacity of the eight tanks is 4,000,000 gallons each 24 hours.

While it is possible to reach this capacity and still secure good results, it is preferable to design a plant with a larger settling capacity per unit of water to be consumed, or, in other words, to design a plant with a larger "settling factor." A safer plan than the preceding, although it involves a greater outlay, and demands greater tank capacity, is to design the plant so that each tank will be emptied three times in 24 hours instead of four. In this way, from 5 to 6 hours will be allowed for settling and from 2 to 3 hours for filling and emptying. These periods are approximate only, and either more or less time than mentioned may be consumed, depending on local conditions, the speed of pumping, and the rate of water consumption. Taking the

rate mentioned, however, and allowing ample time for settling under the worst conditions usually encountered in water softening, the plant under discussion would have a capacity of 1,500,000 gallons each 24 hours. The time required for settling must be looked into carefully, as most supplies vary at different periods, especially if the water is taken from streams that usually carry widely different amounts of suspended matter and dissolved matter at different seasons.

11. Advantages of the Eight-Tank Apparatus. The eight-tank system is perhaps the simplest in arrangement and detail of any system of apparatus, and it is doubtful whether any of the more elaborate designs of either intermittent or continuous plants are more efficient or economical for a long period when all points are taken into consideration. There are no automatic or mechanical devices to get out of order, and each tank has only three valves to control, namely, (1) the raw-water inlet from the water system; (2) the outlet valve for admitting softened water to the feed-line for boilers or other apparatus using the water; and (3) an outlet valve to the sewer for the removal of sludge when it is necessary to clean the tanks. Modifications of this system may be made as desired by increasing or decreasing the number of tanks or their size.

12. Concrete Tanks.—Tanks made of concrete and either rectangular or circular in cross-section are sometimes used to advantage in the eight-tank system. The rectangular tank will be found the best shape to adopt if room is limited. Concrete tanks are generally sunk into the ground, their walls extending above the surface to whatever height is desired or is made necessary by local conditions. Usually, however, they should not extend more than a few feet. The advantage of placing tanks in this way is that the raw water does not have to be pumped so high, and, besides, the retainers require no care or attention. The cost of concrete tanks depends on local conditions, and if the plant is to be permanently located, the tanks will, in the long run, be as

cheap as wooden or steel tanks, or perhaps cheaper. With the growing use of concrete and the probable cheapening of cement, it would seem to be a form of construction that is to find increasing use in water-softening construction in the future, in all large plants at least.

13. We-Fu-Go System.—In Fig. 3 is shown the **We-Fu-Go water-softening apparatus.** This apparatus is

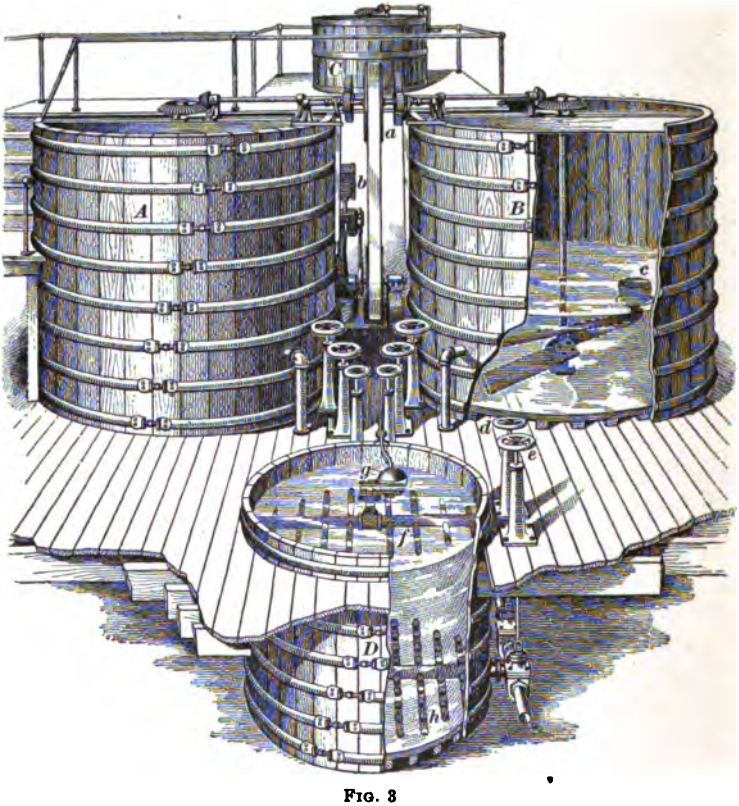


FIG. 3

of the intermittent type and is manufactured by the William B. Scaife & Sons Company, of Pittsburg. It consists of the treating tanks *A* and *B*, a solution tank *C*, and a filter tank *D*. The treating tanks are fitted with stirring devices that con-

sist of paddles attached to a vertical shaft in the center of each tank and are driven by bevel gearing by a horizontal shaft that is geared to a jack-shaft carrying the driven pulley *a*. Driving may be made by an electric motor or by a steam engine *b* belted to the pulley *a*. It is the usual practice to fit clutches to the driving gearing of the stirrers, in order to permit either one to be thrown out of operation. The reagent, or solution, tank *C*, placed on top of the treating tanks, is fitted with a hand-operated stirring device, and has valved pipes, not shown, for discharging the reagent into either of the treating tanks. This solution tank may also be placed on the ground or in any convenient location and the reagent pumped into the treating tanks; this arrangement is preferable, as it saves the labor of handling the reagents. Each treating tank is supplied with a floating discharge pipe *c*, a pipe that admits raw water, and a sludge discharge pipe. Each pipe has its own valve, the wheels of which are placed where most convenient; in the illustration, they are shown on the platform on which the treating tanks stand.

The filter tank *D* is used to aid in the final clarification of the water coming from either of the treating tanks. The treated water enters the top of the tank *D* through the perforated pipes *f*. The discharge from either treating tank into the filter is controlled by a balanced valve operated by the hollow copper float *g*, and the water level in the filter is thus kept constant. The water passes down through a filter bed, consisting of a layer of sand on a layer of coarse gravel, into the underdrain or strainers *h*, and then to its destination. In cleaning the filter, a current of water is forced through the filter under pressure, entering through the strainers *h*. This is done by closing the valve *e* in the pipe that leads the filtered water to its destination and opening the valve *d* in the pipe that contains water under pressure. The accumulated dirt and sludge in the filter bed is washed into a trough that is placed either across the top or around the edges of the filter bed and connected by a pipe with a valve to the sewer.

14. The treating tanks *A* and *B*, Fig. 3, are alternately filled with raw water, and the reagents are added from the solution tank *C*. The reagents are dissolved in the tank *C*, the requisite amount of lime being first slaked and then added while one of the treating tanks is filling. The soda ash is then dissolved in the tank *C*, and allowed to flow into the treating tank when the latter is full or nearly full. The stirrer in the treating tank is started while the tank is filling, and is continued in operation for 15 minutes after the tank is entirely full. In this way, a thorough mixing of the reagents with the water is effected and the sludge, or precipitate, left in the tank from preceding treatments is thoroughly stirred up.

15. For a battery of four treating tanks, one reagent tank can be made to serve. In such a case, the reagent tank must, of course, be connected with each treating tank by means of valved pipes for the delivery of the reagents. One or more filters may be installed, depending on the volume of water to be treated, as well as on the character and quantity of the precipitate to be removed. If the precipitate consists mainly of a crystalline sludge of calcium carbonate, only a small filtering area is required; if, on the other hand, the precipitate is of a slimy character, such as magnesium hydrate or fine silt, a large filtering area will be necessary. Furthermore, if the precipitate, owing to imperfect sedimentation or to too short settling periods in the treating tanks is voluminous, the filter capacity must be of a corresponding size to be able to take care of the extra work expected.

16. If a sufficient interval can be allowed between the time of the addition of the reagents, stirring, and the withdrawing of the softened water, the filter may be dispensed with. This, however, is not to be recommended and is poor economy. The use of a filter permits shorter settling periods, and this means a smaller ratio of tank capacity to the amount of softened water required. The usual ratio of tank capacity is eight times the hourly consumption of

softened water. As an illustration, if the hourly consumption of water is 10,000 gallons, a tank capacity of 80,000 gallons should be provided. Such a plant, requiring 10,000 gallons of softened water per hour, could be arranged with two tanks of a capacity of 40,000 gallons each. This arrangement will allow 4 hours for filling and settling in each tank. Allowing 1 hour for filling and stirring for each tank, which is about the average time, 3 hours will be left for settling.

Experience has shown that 4 hours is the minimum time to be allowed in each treating tank. Except in very rare cases, the reactions are not completed in less time, nor is the water really fit for boiler-feed use unless it has been in the treating tank at least 4 hours. Occasionally, 6 hours or more has to be allowed for each treating tank, depending on the nature and source of the water. In all cases, the time allowed for the treatment of the water should be based on the speed at which the reactions take place and the manner in which the water settles.

17. Importance of Sludge.—The presence of sludge in the treating tanks from previous treatments is of great importance. In early experiments with water-softening apparatus, it was learned that if any sludge remained in the treating tanks and was stirred up after the addition of a new supply of treated water, the new precipitate would be readily carried down by the sludge when resettling. The action of the sludge, although purely mechanical, thus greatly helps the precipitation and settling processes. There is practically no limit to the amount of old sludge that may be left in the treating tanks, so far as the rate of settling is concerned. There is a limitation, however, set by its volume in the tanks, as with water carrying a large amount of silt and mud or precipitating a bulky carbonate, the accumulated sludge soon begins to interfere with the stirring device; or, if floating discharge pipes are not employed, there is always danger that the sludge, if greatly in excess, will be carried through the discharge outlet of the tank with the softened feedwater.

With average waters, it is necessary to remove the sludge at intervals of 1 to 4 weeks, and with waters producing a very voluminous precipitate, the sludge must be removed oftener. No hard-and-fast rules can be laid down, however, as each individual water creates its own rule, based on the analysis of the water and on actual experience. In all cases, the sludge should never be entirely removed; a portion of it should be left in the treating tank in order to facilitate the settling of the first unit of water treated in a cleaned tank.

It should be understood that these remarks about sludge apply not only to the We-Fu-Go type of apparatus, but to all types of intermittent apparatus that depend on sedimentation.

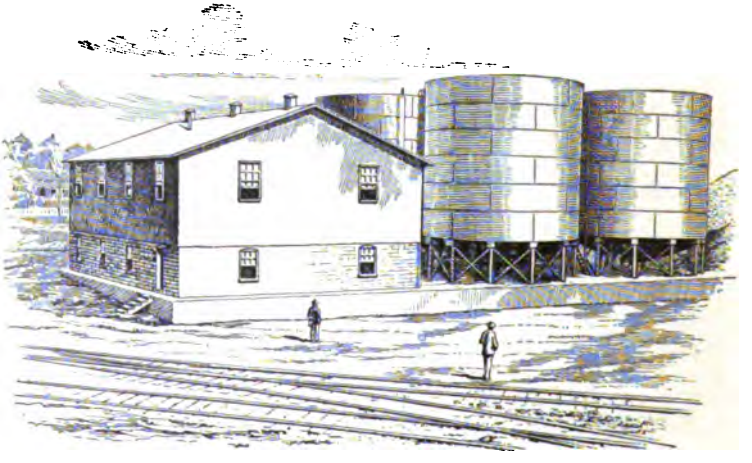
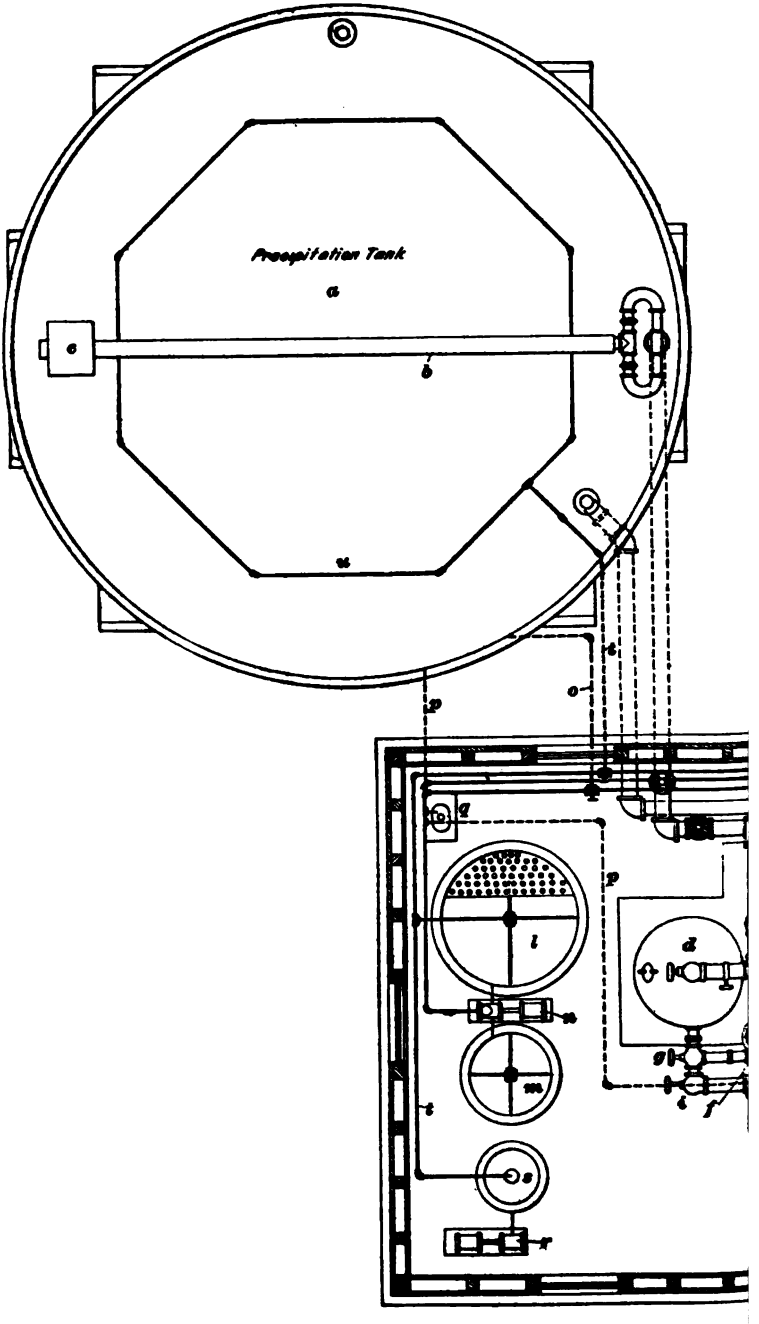


FIG. 5

18. Pittsburg Filter Manufacturing Company's Intermittent Apparatus.—In Fig. 4 is shown a diagrammatic top view and in Fig. 5 a perspective view of an intermittent water-softening plant designed by the Pittsburg Filter Manufacturing Company, of Pittsburg. While all intermittent plants are, of course, very similar in construction, this one differs from all others in some important details.

Referring to Fig. 4, the plant shown consists of two large treating tanks *a* fitted with floating outlet pipes *b*, having


Precipitation Tank
a
b
c
d
e
f
g
h
i
k
l
m
n
o
p
q
r
s

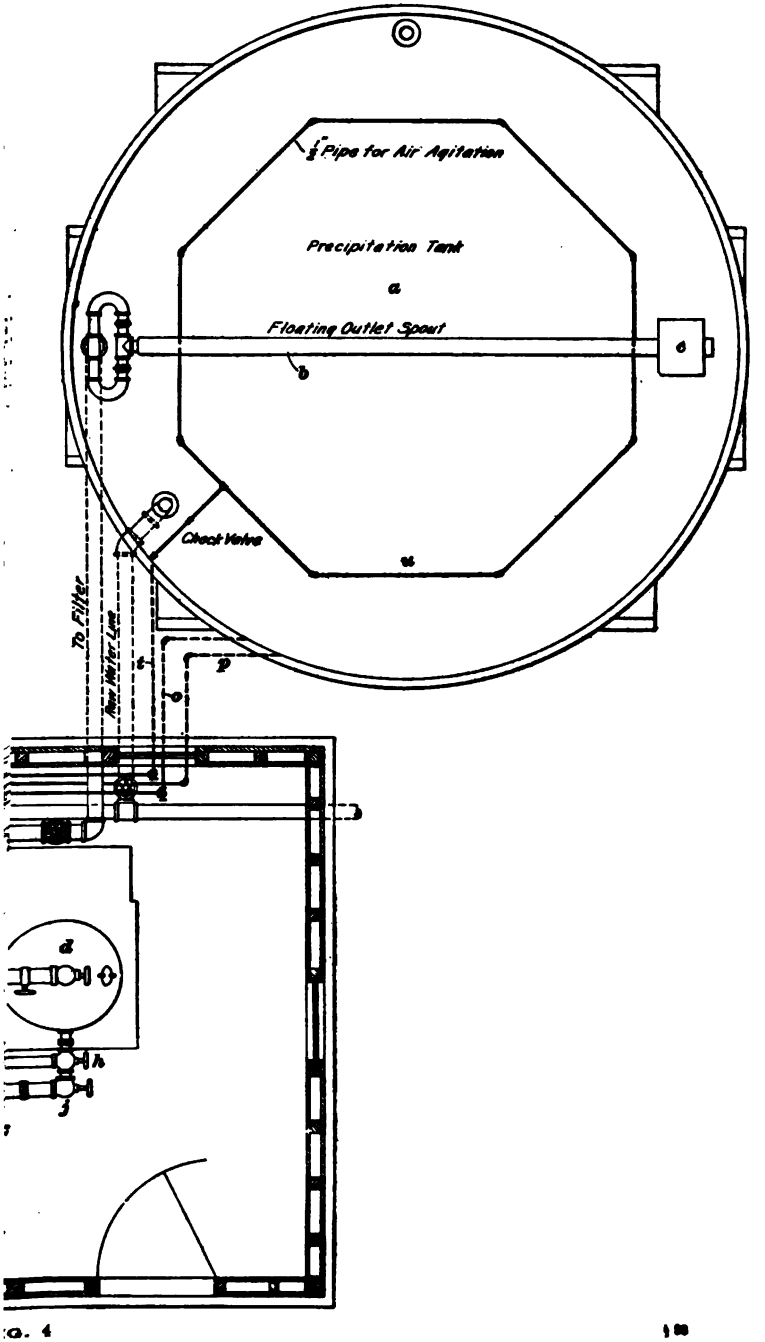

½" Pipe for Air Agitation
Precipitation Tank
a
Floating Outlet Spout
b
c
Check Valve
e
To Filter
Raw Water Line
f
g
d
h
j

Fig. 4

floats *c*. The outlet pipes *b* connect with two filtering tanks *d* that are placed in a separate building, in which are also placed the solution tanks. The piping is so arranged and valved that both treating tanks can be discharged simultaneously to both filter tanks, or that either one of the treating tanks can discharge into either one of the filter tanks. A by-pass connection *e* provided with a valve, not shown, allows the filters to be cut out; that is, permits the treated water to pass directly to its destination without filtration. The filtered water is discharged from the bottom of the filter tanks into the pipe *f*. The tanks can be emptied through the proper manipulation of the valves *g*, *h*, *i*, and *j*, of which *g* and *h* are three-way valves and *i* and *j* ordinary angle valves. Thus, supposing that the left filter tank is to be put into use, the valves *h* and *i* are closed and the valves *g* and *j*, as well as the valves admitting the purified water, are opened. The valves admitting the treated water to the right filter tank are of course also closed. By opening the valve *j* the right-hand filter tank can be emptied for the purpose of cleaning through the pipe *k* into the sewer, and by opening the valve *i* the left-hand filter tank can be emptied through the same pipe.

The tanks *l* and *m*, which are housed in the same building as the filter tanks *d*, serve as receptacles for the reagents; these are forced into either of the treating tanks *a* by a small pump *n* through the pipes *o*. The pipes *p* lead from the treating tanks to a basin *g*. The object of these pipes is to convey the treated water to the basin, from which it can be conveniently removed to be tested in order to ascertain whether sufficient quantities of the reagents have been added.

Agitation of the water in the treating tanks is accomplished by compressed air furnished by an air compressor *r* to a storage tank or receiver *s* and conveyed through the pipes *t* to an agitator *u* made of perforated ½-inch pipe.

19. As shown in Fig. 5, the bottoms of the tanks made by the Pittsburg Filter Manufacturing Company are cone-shaped. Such bottoms are called *sludge bottoms*. At the

apex of each base is placed a valve, through which the sludge may be discharged from the tank. The advantage of this feature is that the sludge will collect in this conical bottom and the treated water may be withdrawn to the top of the cone. In this way approximately 95 per cent. of the total contents may be withdrawn, whereas only about 80 to 85 per cent. of the total contents of an ordinary flat-bottomed treating tank is available. The available tank capacity is thus increased about 10 to 15 per cent. A further advantage is that in flushing out sludge, a large volume of softened water is saved, amounting to 1 or $1\frac{1}{2}$ per cent. of the total volume treated. With flat-bottomed tanks, nearly one-fifth the contents is left in each time the treated water is drawn off, and this is lost each time the tanks are emptied to flush out the sludge. Also, less time is required to remove sludge from tanks having cone-shaped bottoms than from tanks with flat bottoms.

As further shown in Fig. 5, the tanks are of steel construction and are supported on columns suitably braced for stiffness. These columns are 8 feet high, thus affording ample space in the inverted-cone bottom for the collection and removal of sludge. Stairs and platforms are located between the tanks, to give ready access to them. The tanks shown are 30 feet in diameter and 30 feet high, not considering the cone-shaped bottom, giving an available capacity of about 160,000 United States gallons. The total capacity is some 10,000 gallons greater, but as this extra amount of water is not withdrawn when the tanks are emptied, each tank has the available, or effective, capacity just mentioned.

20. In operating the Pittsburg Filter Manufacturing Company's intermittent apparatus, the predetermined amounts of reagents, as lime and soda ash, for instance, having been weighed out and dissolved in the corresponding tanks, the milk of lime or lime water, as the case may be, is forced by the pump into the large treating tank while filling. When the tank is nearly full, the same pump introduces the soda-ash solution. In the meantime, the air is turned on to mix

the water and the reagents; the agitation is continued for 15 to 30 minutes and the precipitate allowed to settle. The interval for settling depends on several conditions, as has been previously mentioned, but 2 hours is generally enough. This period is determined in most cases by the demand for water, and depends on the ratio of tank capacity to consumption. Reagents are constantly prepared in the pair of tanks *l* and *m*, Fig. 4, and are always ready for pumping into the large tanks as they are emptied and refilled. Each tank of water is tested after the reagents are added. The testing is readily and conveniently done by withdrawing some of the water through the pipe *p* to test basin *g*. If an insufficient amount of reagents has been added, a correction may be made and the air turned on to mix the water again; if, on the other hand, the tests show an excess of the reagents some of the water may be run off and the tank filled with raw water. The ease with which corrections can be made is an apparent advantage of any intermittent system over a continuous one, but with care and intelligent control, corrections seldom have to be made, even with rapidly changing river or creek waters.

21. Comparison of the We-Fu-Go and Pittsburg Filter Manufacturing Company's Systems.—The apparent and chief difference between the We-Fu-Go and the Pittsburg Filter Manufacturing Company's systems is that in the former the reagents are taken to the top of the tank and the solutions made there, whence they are added direct into the treating tanks without the aid of pumps and piping, while in the latter, the reagents are prepared in the operating house on the ground level and pumped into the large settling tanks. In this arrangement, all reagent tanks, filter tanks, etc., are assembled in one group, and the plant is therefore very convenient for operation. It will not be necessary to discuss here the merits of either arrangement. Each system has certain points in its favor, and both have a wide and extended use with splendid records of success and efficiency. The mixing by means of a stirring device or by means of air agitation may be regarded as equally efficient.

The general arrangements described and illustrated, with more or less slight changes and modifications, are made use of by all manufacturers of intermittent apparatus. The two types discussed have perhaps the most extensive use, and are good examples of intermittent softening plants as generally constructed today.

DESCRIPTION OF CONTINUOUS APPARATUS

22. As has been previously stated, the continuous system of softening water was developed later than the intermittent, and it may be said that the continuous apparatus has only within very recent years reached a development thoroughly adapting it to the work of water softening. While some forms of continuous apparatus have been in use for more than 30 years, improvements have been gradual, and many defects had to be eliminated before the operation of the apparatus became satisfactory.

In the continuous system, as the name would imply, a continuous supply of raw water goes to the apparatus and soft water is continuously produced, or, in other words, the water is softened as it flows through the apparatus. The points in favor of this system have been enumerated, and it need only be repeated that so far as quality of product or efficiency of softening is concerned, there is very little choice between the continuous and intermittent systems for the majority of conditions.

The following descriptions and illustrations of continuous apparatus are not given necessarily in the order of their development, but simply to illustrate the various types. The devices that are in use today on some of the best known apparatuses are either exactly the same as earlier ones or merely modifications of the earlier forms.

23. Continuous Lime Saturater.—The device known as a **continuous lime saturater** was quite early applied in continuous water softening, and it is a prominent feature of all but one or two types of continuous apparatus in gen-

eral use today. It is essentially a tall, narrow cylinder, in the lower part of which lime, *CaO*, is placed, and over and through which water for slaking is made to flow continuously at such a rate as to insure complete saturation. The cylinder is, or should be, of sufficient height and area that a perfectly clear and saturated lime-water solution will collect at the top. Many of the devices in use have been defective in producing irregular lime water; that is, after a fresh lime charge was introduced, the first lime water received would be fully saturated, but before the lime was nearly exhausted, the lime water would be only partly saturated. The irregularity of lime treatment thus produced is objectionable in all cases, and under certain conditions may cause very serious trouble.

In order to insure the complete saturation of the water and a complete exhaustion of the lime, long and careful investigations have been carried on and various schemes for agitation have been devised. In some, the mixture is agitated by mechanical stirrers, driven by different means. In others, agitation is accomplished by blowing air or steam into the lower part of the saturater. Steam, by the way, has never proved a success, as the solubility of the lime decreases with an increase in temperature. As a rule, in a properly designed saturater, no agitation other than that produced by the influx of the water is necessary.

Many modifications of the saturater are used in different types of apparatus, some of which are shown in the illustrations of continuous apparatus. To the Austrian engineers Elbel and Siegmuth is given the credit of constructing the first lime saturater, which consists of a cylinder for saturating the water. This saturater, as previously mentioned, allows the water to percolate upwards through the lime slowly, the milky liquid gradually clearing and emerging from the top of the cylinder as a clear and saturated lime-water solution. While credit for the first continuous saturater is believed to be correctly given, it is thought to have been first applied in the Dervaux softener, a Belgian invention.

24. Dervaux Continuous Apparatus.—The **Dervaux continuous apparatus** is shown in Fig. 6. It consists essentially of a lime saturater, tanks for preparing the soda-ash solution and cream of lime, and a settling tank or

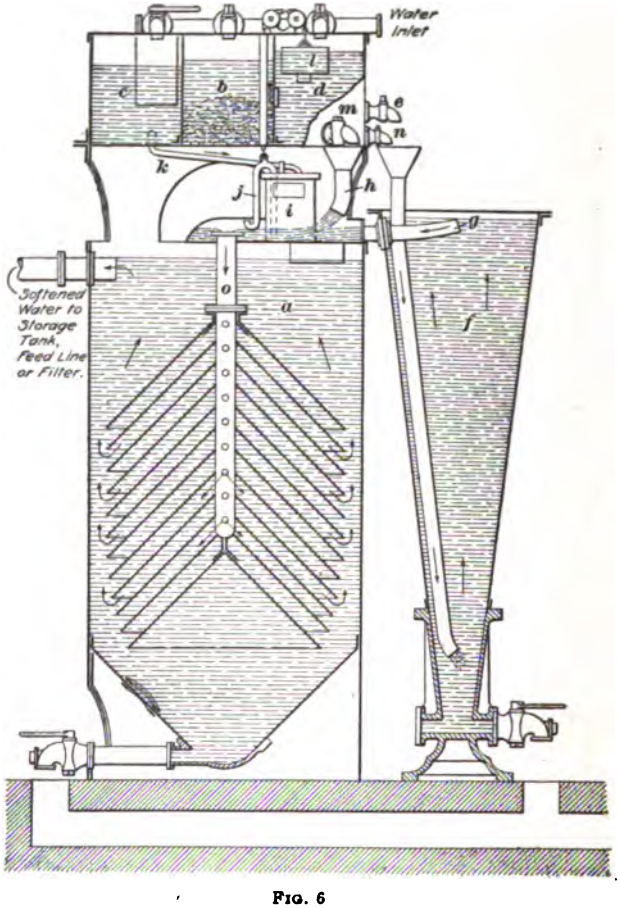


FIG. 6

reservoir. It may be operated with or without a separate filter. In the settling reservoir, or clearing chamber *a*, are cone-shaped plates placed one above the other. On these plates the precipitated matter lodges, and after it has built up to a certain height, it gradually slides off and collects in

the conical bottom of the tank, from which it is washed out at given intervals. The height of the settling reservoir *a* may be increased and an excelsior filter placed near the top to catch the lighter particles that are not removed as the water rises in the settler; or, any other arrangement of placing the filter can be made to suit local conditions or personal preference. Tanks *b* and *c* for preparing the reagents are located above the settler *a*, and raw water enters the tank *d*. These tanks are connected by pipes, with valves to control the admission of reagents and raw water. Lime is slaked in tank *b*, and the cream-of-lime paste is admitted through valve *e* to the lime saturater *f*, a pipe carrying it nearly to the bottom. The water on rising gives a saturated solution of lime water for admission through pipe *g* to meet the raw water as it comes through *h*, and the solution of soda ash from *i* is delivered through the siphon *j*, which reaches to the bottom of *i*.

25. The operation of the Dervaux apparatus is as follows: The soda-ash solution prepared in *c* flows through the small pipe *k* to the box *i*, from which the siphon *j*, connected by a chain to a float *l* in the raw-water box *d*, delivers the soda-ash solution to the incoming raw water. The lowering of the level of the water in *d* below the discharge pipe causes the float to lower and thus raises the siphon in *i*, so that the flow of the soda-ash solution stops simultaneously with that of the raw water. The valves *m* and *n* on the raw-water box *d* deliver the raw water to the lime saturater *f* and to *h*, respectively, and as both are at the same level, the supply of raw water is automatically stopped as soon as the supply of soda-ash solution is cut off. With a supply in *d*, the float rises, the siphon *j* drops back into *i*, and the apparatus is put into operation again automatically.

The working of the apparatus will now be readily understood. The water enters at *d* and leaves at *m* and *n*. That part of the water which leaves through *n* goes nearly to the bottom of *f*, becomes saturated with lime, and, together with the raw water from *m* and the soda-ash solution from *j*,

finds its way, as indicated by arrows, through perforations in pipe *o* to the settling tank *a*, where it rises and leaves, as indicated.

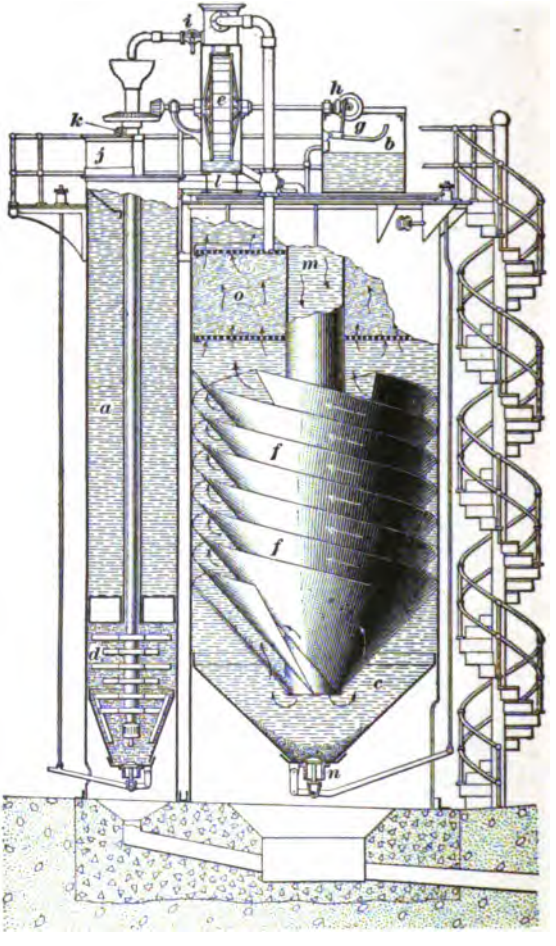


FIG. 7

The apparatus is built with certain modifications, but essentially as illustrated. The lime box *b* is sometimes dispensed with, and the lime added direct to the saturater, which may be changed sometimes to have a scheme for

measuring the lime water and various arrangements of the tank and piping for delivery of the reagents.

The Dervaux apparatus is used extensively in France and Germany. It is described here because some of the features believed to have been first applied in this apparatus are prominent points, though modified, in some of the recent and best American apparatus.

26. Desrumaux Continuous Apparatus.—A sectional view of another continuous apparatus known as the **Desrumaux** is shown in Fig. 7. It consists essentially of the same three parts as the Dervaux apparatus, namely, the continuous lime saturater *a*, the soda reagent tank *b*, and the settling tank *c*. The chief difference between these two types of apparatus is the method of control in adding reagents by the use of a waterwheel. The illustration shows clearly the minor differences in the lime saturater, which has a stirrer *d* driven by a waterwheel *e* on top of the tank, and the arrangement of settling plates in a series of spiral cones *f* instead of inclined plates as in the Dervaux apparatus. In later adaptions of the Desrumaux apparatus, the spiral cones have been abandoned. The soda reagent tank *b* is fitted with a scoop *g* that is operated from the waterwheel by the bevel gearing *h*. This device scoops up a definite amount of solution at each revolution, and as the supply of raw water over the wheel varies, the number of lifts of the scoops in the soda tank varies, thus automatically proportioning the feed of soda solution to the volume of raw water. A valve *i* over the waterwheel serves to proportion the volume of water fed to the lime saturater. Instead of the scoop or spoon device shown for proportioning the soda that is to be added, this operation is sometimes controlled by a float located in the inlet water box over the wheel and connected by a chain to another float in the soda tank.

27. In operating the Desrumaux apparatus, the lime is slaked in the tank *j* and then passes to the bottom of the saturater *a*. The quantity of water passing to the lime saturater depends on the lime required to soften the water,

and is controlled by the valve *k*. The remainder of the water passes over the waterwheel, which drives the stirrer in the lime saturater and the proportioning device, spoon, or scoop (provided the chain and special float are not used). The rate of pumping, or flow, to the apparatus, as well as the water needed to dissolve the required amount of lime, being known, the soda-ash solution is made of a convenient strength to last a certain interval. As the raw water flows to the apparatus, the operations are automatic as long as the reagents last in each tank, and with their renewal, the operations continue indefinitely. The lime water overflows from the saturater through the pipe *l* and mixes with the water that has passed over the wheel and also the soda solution. It then passes down the central pipe *m*, where a more intimate mixture occurs and the softening reactions are practically completed. Reaching the bottom of the pipe *m*, it passes upwards through the spiral settling plates *f*. These plates retain the bulk of the precipitated matter, which gradually slides off and collects on the conical bottom of the tank, from which it is discharged through valve *n* to the sewer. At the top of the tank, the water passes through an excelsior filter *o*, which effects the final clarification.

28. In the continuous apparatus described, as well as in many others, the principles of change in direction of flow and of diminished velocity as the water emerges from the central downtake to the enlarged area of the settling chamber, are of the utmost importance in the efficiency of sedimentation and the softening of the water. The importance of these principles has been already referred to in connection with what is believed to be the first continuous apparatus. It is of interest to note how firmly this principle, early recognized and applied in continuous apparatus, has remained an important feature of all, and how much it is made use of today, with no great variation, in most continuous appliances.

29. Brunn-Loewener Continuous Apparatus.—The essential feature of the **Brunn-Loewener system** is an oscillating bucket having two compartments. This oscil-

lating, or tipping, bucket serves the purpose of the water-wheel shown in Fig. 7, and is made to operate a stirrer in a reagent tank placed above the bucket and also to operate a valve to discharge automatically the proper amount of the mixture of milk of lime and soda ash. The device as applied to water softening was originally patented by Brunn, in Denmark, but at present it is known in the United States as the **American water softener**, and is controlled and built by a company of that name.

30. In the Brunn-Loewener apparatus, the solutions are prepared on the ground level and pumped to the chemical tank on top of the large settling tank or tower, from which they are delivered to the raw water in the tipping bucket. Fig. 8 (*a*) shows a tank *A* in which sufficient lime is slaked to operate for 12 hours. The milk of lime passes through a screen to the lower tank *B*, to which the required amount of soda ash is added. This mixture is kept thoroughly stirred by an agitator *a*, which is driven by the water motor *C* by chain and sprocket wheels. The same shaft that drives the agitator also operates the pump *b* that raises the solution to the chemical tank on top of the main tank. The motor furnishing the power is driven by the incoming water, a pressure of a few pounds being sufficient.

The raw water first passes through the motor and then up the pipe *c*, to the oscillating bucket *d*, Fig. 8 (*b*), where the measuring and mixing takes place. The raw water delivered through the pipe *c* runs into one chamber of the oscillating bucket until the bucket tips and dumps the water into the large tank. At the same time, the other compartment of the bucket comes underneath the raw water, and the same operation is repeated. Thus, alternate filling and dumping is continued as long as the raw water is discharged from *c*.

Above the bucket is a semicircular tank *e* containing the mixture of reagents pumped from the tank *B*, Fig. 8 (*a*), through pipe *f*. In the bottom of the tank *e*, Fig. 8 (*b*), is a valve through which the solution passes into the oscillating bucket. This valve is operated by the rod *g* and counter-

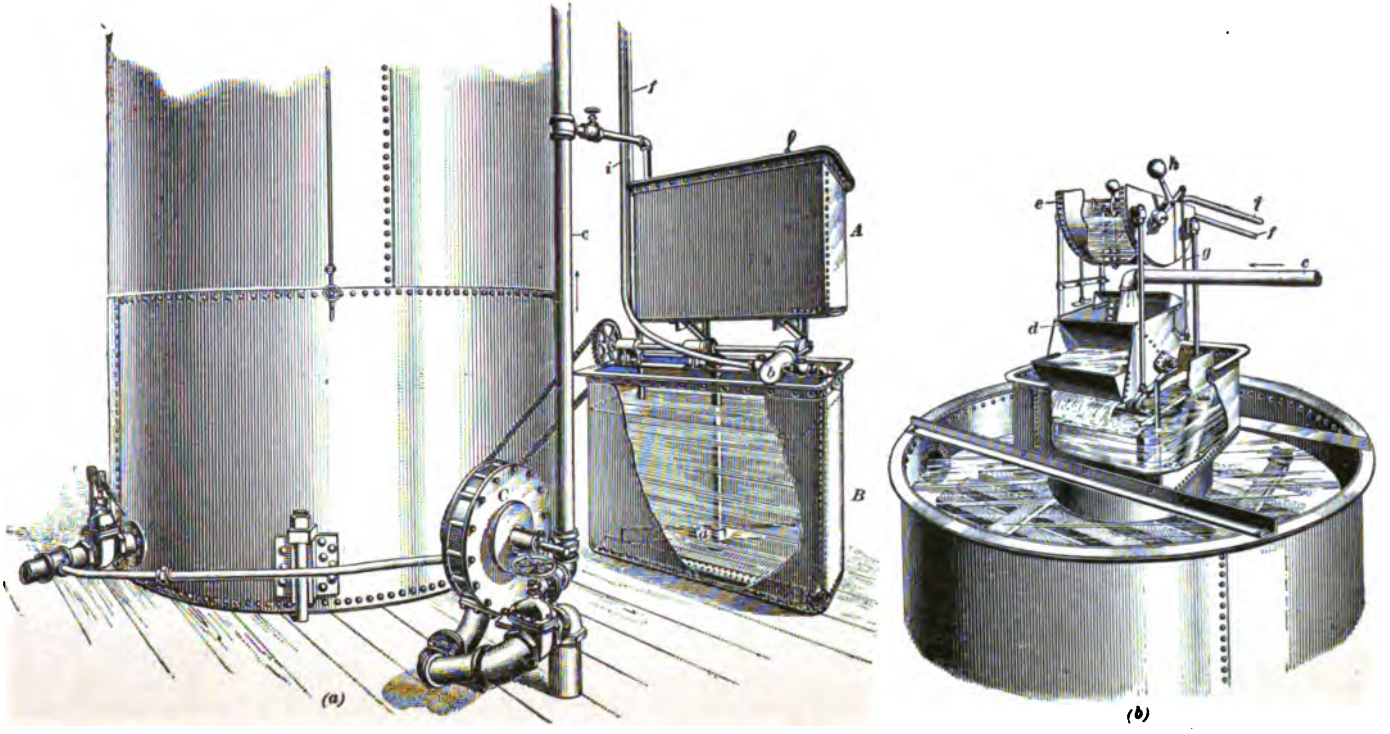


FIG. 8

weight *h*, being thrown open by each oscillation of the bucket, or *tipper*, as it is generally called. It thus delivers a definite and constant amount of the reagents to each bucket of water, and is closed automatically by the next oscillation. The lift of the valve, however, is adjustable, so that the valve opening is easily regulated and the amount of solution delivered to the bucket readily adjusted as conditions of the water may require.

31. The tipping bucket is always moved by exactly the same volume of water, without regard to the rate of flow or the number of oscillations, and the valve in tank *e* being adjusted to deliver the amount of reagents required, this quantity is automatically and accurately discharged to each bucketful of raw water. The delivery of reagents is further controlled by keeping the depth of the reagents solution in tank *e* constant. This is governed by an overflow pipe *i*, which returns the reagents delivered through the pipe *f*, in excess of that passing through the valve to the tipper. The regular head thus provided always feeds the exact amount of reagents determined by the valve setting. A stirrer in tank *e*, also operated by the tipper, keeps the mixture of the reagents uniform.

On leaving the oscillating bucket, the water passes to a compartment above the central downtake *j*, Fig. 9. A deflector plate is so set therein as to give the water a whirling motion as it enters the inner pipe. This motion mixes the raw water more thoroughly with the reagents. It is claimed that this motion also causes a more rapid coagulation of the precipitate and a better settling in the larger area of the uptake, so that less work is thrown on the filter. The arrows show the direction of the flow, and at the top is shown the filter. The filter, which is made of excelsior, or some similar material, removes any sludge that does not settle out in the uptake. From here, the water passes to the storage tank through the outlet pipe.

32. The Brunn-Loewener, or American, water softener has some of the points common to many continuous softeners,

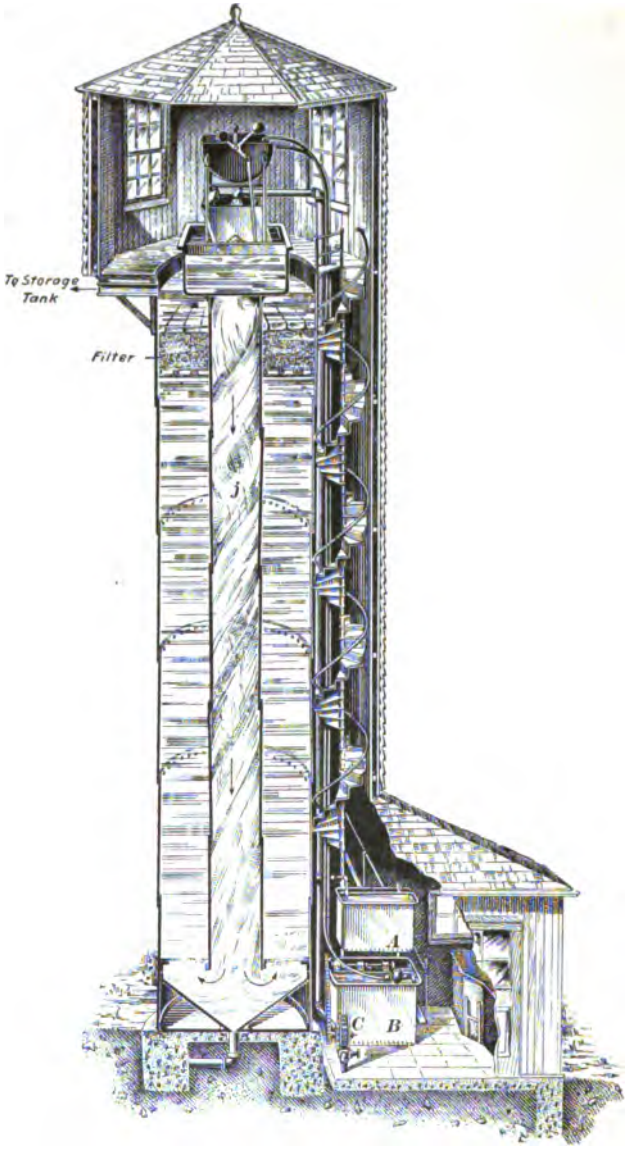


FIG. 9

such as the central downtake, the enlarged uptake where settling and clarification takes place, and the filter at the top.

In addition to the oscillating bucket as the principal variation, a 10-per-cent. solution of milk of lime is used in this system instead of a lime saturater or some other device for giving a large volume of saturated lime water. The obvious advantage is the smaller lime tank, which is only about one-eightieth the size required for lime water. It is claimed that a more constant composition is secured in this way, but this would seem to be open to question unless the lime used was of unusually regular composition. Both reagents are prepared at ground level and pumped to the top of the apparatus. Although this is not distinctive of the Brunn-Loewener apparatus, as other continuous and intermittent types also elevate the reagents, it was probably the first so designed.

33. Davidson's Continuous Water Softener.—The **Davidson water softener** utilizes a tipping bucket for measuring the raw water and proportioning the addition of reagents. As the bucket tips it operates a stirrer in the chemical tank (similar to that in the Brunn-Loewener apparatus) and two pumps that force into the water in the tipper the calculated amount of reagents to be delivered. The stroke of the pumps can be adjusted so as to deliver varying amounts of the chemical solution. The water from the oscillating bucket is led to the bottom of one of the settling tanks, to mix with and stir up the old sludge and thus hasten sedimentation. The steam pump used to pump the water up to the tipping bucket is also utilized to mix the old precipitate on the bottom of the tanks with each new tank of water. For this purpose, perforated pipes are placed in the bottom of the tanks. Two settling tanks are usually provided, the contents of one settling while the other is filling. Floating discharge pipes are used to withdraw the softened water from the surface.

This system was developed by G. M. Davidson, Chemist and Engineer of Tests of the Chicago and Northwestern

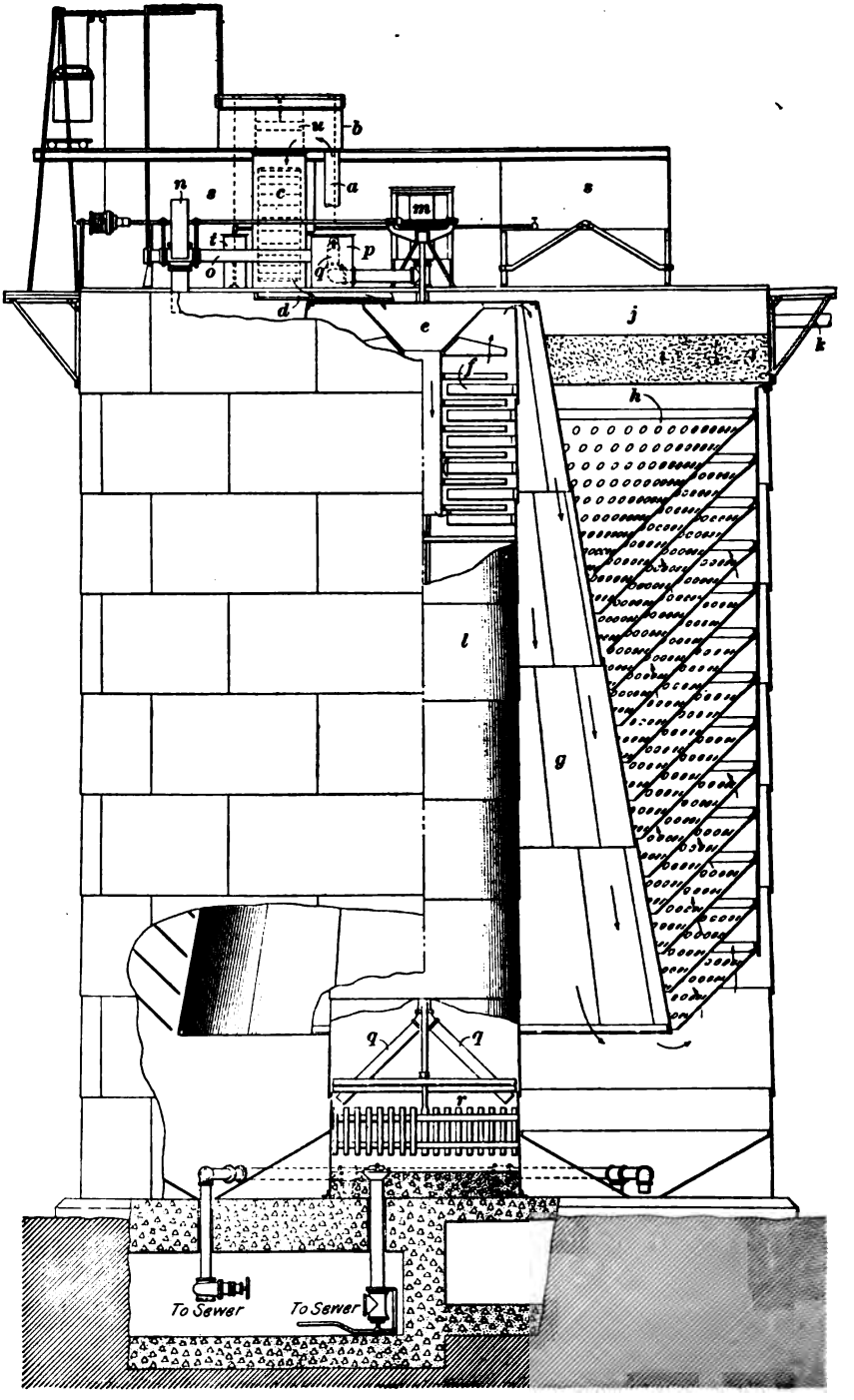


FIG. 10

Railroad Company, and is extensively used by that railroad for softening water for locomotive use. This apparatus combines the features of the intermittent and continuous types. It has two settling tanks, which are alternately filled and emptied after the sludge has settled. The method of adding the softening solution, however, is continuous, and as this is the novel feature of the system, it is generally classed as a continuous apparatus.

34. Kennicott Continuous Apparatus.—The **Kennicott system of water softening** has the most extensive use of any continuous apparatus in the United States, particularly in the railroad field, and has given efficient service and excellent results. It has most of the features of earlier continuous apparatus, but many of them have been developed and improved, namely, the feed-proportioning device for reagents, the lime saturater, which is placed concentrically in the structure, the use of soft water for lime water, variation in the central downtake, etc.

35. A diagrammatic view of the Kennicott apparatus, which also shows the method of operation, is shown in Fig. 10. The water is pumped up through the pipe *a* into box *b* above the waterwheel *c*, to which it is delivered through a slot in the bottom of *b*; it then passes through the pipe *d* into the mixing chamber *e*, where the lime and soda-ash solutions are added. The water and reagents now come in contact with the revolving deflector plates *f*, and are more intimately mixed and stirred. The water then turns upwards and overflows into the downtake, or settling cone *g*, which is of continually increasing diameter toward the bottom, so that the velocity of the flow decreases, thus permitting the greater part of the precipitate to settle out by the time the downward current reaches the bottom of the cone. Here, the flow is reversed again and the water ascends through a series of conical baffle plates *h*. The velocity of the ascending water is again decreased, owing to the enlarging area of the upper passage, as shown in the illustration. The plates catch most of the remaining precipitate, which finally slides

off to the tank bottom to be discharged to the sewer. A wood-fiber filter *i* at the top completes the removal of the sludge, and the softened water passes to a shallow tank *j*, from which it is delivered through the pipe *k* to the storage tank. The water may be withdrawn from *j* at any point or number of points to as many storage tanks.

The lime-water solution is prepared in the continuous saturater *l*, which is placed inside of the settling tank to

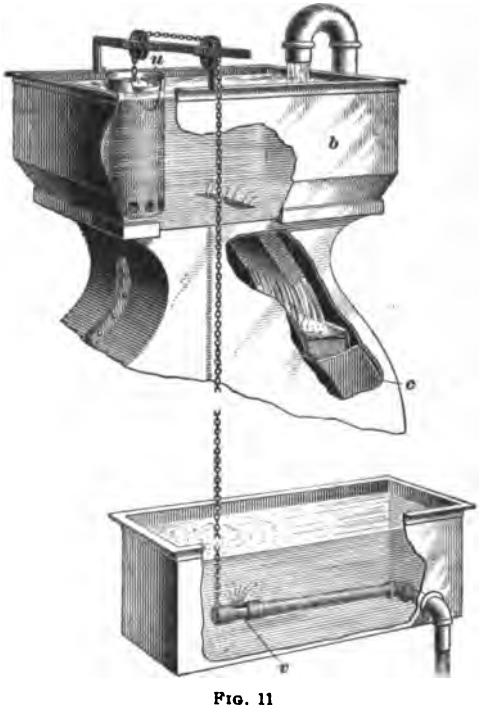


FIG. 11

prevent freezing and to assist in keeping the water at a more nearly uniform temperature, thus giving a more regular lime water. The lime is slaked in a box *m* directly over the saturater. Soft water for slaking is raised by a wheel *n* having hollow curved arms that connect with the hollow shaft *o*, and then flows to the tank *p*, where its flow to the saturater is controlled by a hinged regulating pipe *q*.

The soft water enters through the pipe *q* to the lime box *p* and passes to the bottom of the saturater, where the stirrer *r* keeps it thoroughly mixed. The solution rises in *l*, and the excess of lime gradually settles out until a clear saturated lime water enters the mixing chamber *e*. Here it is thoroughly agitated with the raw water and soda-ash solution. The soda ash is placed in wire baskets in the two tanks *s*, which fill the soda-solution tank *t* by gravity, the supply of water to the tanks *s* being controlled by the usual valves and floats.

36. The device for proportioning the feed of reagents, spoken of as one of the features of the apparatus, is shown in Fig. 11, where the letters correspond to those in Fig. 10. At *u* is shown a float with a guiding cylinder in the raw-water box *b*. From the float chains pass over pulleys to the swinging pipe in tank *t* (occasionally, two tanks are used). The end of this pipe is slotted and is provided with a threaded sleeve, so that the size of the opening is adjustable to the amount of reagent required. The flow of reagent, with a given orifice, is controlled by the head of water above the orifice. As the chemicals must be added to the raw water in proportion to the amount entering the softener, which depends on the head of water in tank *b*, the latter must be made to control the head of water above the orifice of pipe *v* in tank *t*. As the head in *b* increases, the float *u* rises and the slotted pipe *v* is lowered in the reagent tank, thus increasing the head over the orifice, or slot, and the flow through it correspondingly; with a lessened head in *b*, the float lowers and the slotted pipe in *t* is raised, decreasing the flow of the reagent. The same principle is made use of in controlling both lime water in tank *p* and soda ash in tank *t*, and very close adjustment is obtained with the additional aid of the adjustable screw sleeve.

The use of soft water for preparing lime water avoids the precipitation of calcium-carbonate sludge in the saturater. With waters high in bicarbonates, this sludge is sometimes bulky enough to foul the saturater.

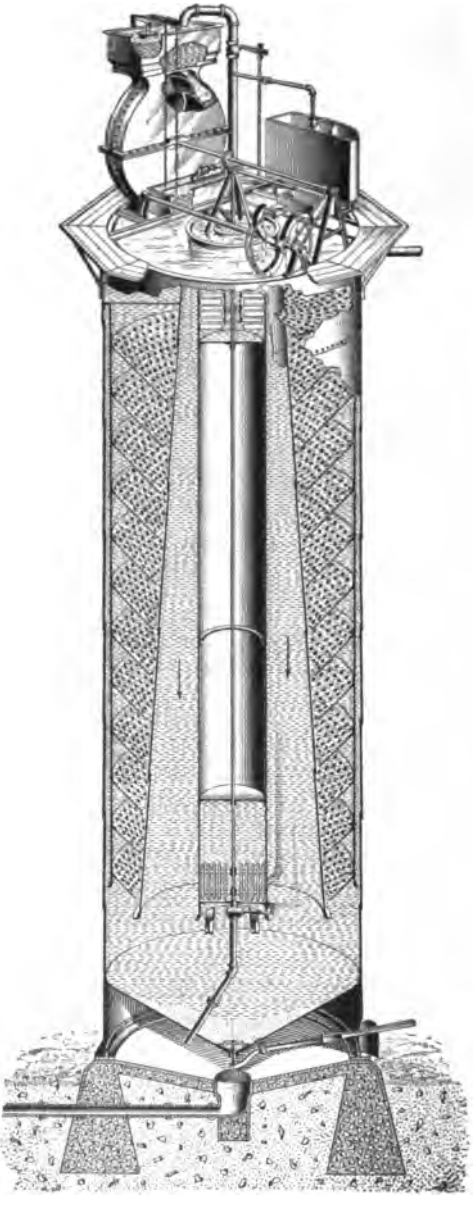


Fig. 12

The wheel that lifts the soft water to slake the lime is driven by a chain and sprocket from the shaft of the main wheel *c*, of the overshot type, and furnishes power for this and the operation of a hoist to raise the reagents to the top of the tank.

37. In Fig. 12 is shown a perspective sectional view of the Kennicott apparatus that illustrates the internal arrangement very clearly. Suitable housing protects the parts on top of the tank from the weather. The apparatus is built in sizes having capacities that vary from 10,000 to 150,000 gallons per hour. To suit local conditions, changes may be made, but the apparatus is always constructed essentially as shown and described. Additional reagent tanks may be provided if other chemicals have to be used or if a coagulant for assisting sedimentation appears desirable.

BOILER COMPOUNDS

DEFINITION AND CLASSIFICATION

38. Boiler compounds are to be regarded as palliatives only. They seldom do more than mitigate bad water conditions when the treatment of the water outside the boiler is not to be secured. Obviously, a boiler is not intended for a softening apparatus, but should be furnished with suitable water so that its own special duty may be fulfilled to the best advantage. There are many times, however, when it is necessary to use boiler compounds or have no softening whatever. This is particularly true of small plants that are handicapped with bad boiler waters, as most large plants are willing to take up the matter of treating their supply, provided it is bad.

It is understood, of course, that boiler compounds are mainly reagents introduced into the feed to prevent or reduce scale formation or corrosion within the boiler, without opportunity for the softening reactions to take place or

the removal of the sludge resulting from such reaction, before introduction into the boiler. Some of the compounds do not come strictly under the head of reagents, as their action is more mechanical.

39. Boiler compounds may be classified as (1) those which attack scale-producing material chemically and, acting as reagents, combine with the scale-forming salts and produce a sludge with all or part of the scale-forming salts broken up; (2) those which act mechanically on the precipitated scale-forming material after it has been produced in the boiler, but before it has had sufficient time to form a hard and adherent scale on the tubes and sheets; and (3) those which act both mechanically and as a solvent, the latter action either partly dissolving scale already formed or "rotting" it and leaving it in condition for easy removal.

40. With boiler compounds of the first group, the supposition is that the precipitate will not form into a hard scale and will be readily removed by blowing off at intervals, or that any scale formed will be easily removed with the scaling or cleaning tools.

Boiler compound of this kind may be composed of any of the regular water-softening chemicals. The principal chemical and, in fact, the one found in most all compounds is *soda ash*. The action of this compound is the same in the boiler as in water softening. It neutralizes any free acids or breaks up sulphates, forming sulphate of soda and a carbonate of the element combined as sulphate in the original water. *Caustic soda* is sometimes used, either alone or in combination with soda ash. It is not used so commonly as soda ash, however, on account of its higher price. Owing to its caustic nature and to the readiness with which it takes up moisture and becomes carbonated by the carbonic acid of the atmosphere, caustic soda is also extremely difficult to handle. An excess of caustic soda is also more objectionable than an equal excess of carbonate of soda, as the former foams almost as readily and has a decidedly injurious action on brass valves and fittings.

Another soda salt that has had an extensive use as a boiler compound of the first group is *trisodium phosphate*, $Na_3PO_4 \cdot 12H_2O$. At present, it is not used so much in many sections as it was a few years ago, but it still has quite an extensive application. Its action has been previously explained and need not be referred to here. It is in many respects one of the safest and most efficient "softeners" to use direct in the boiler, although to remove a given amount of lime, it is more expensive than either soda ash or caustic soda.

Another class of compounds belonging to the first group and one that has very wide use in some localities is *tannin compounds*. These compounds are obtained from vegetable sources, such as sumac, gallnuts, and various barks, and are generally combined with soda, forming tannate of soda. Their action is on the lime and magnesia salts, forming sodium sulphate or carbonate and tannate of lime or magnesia as a light flocculent precipitate, which is either blown out the same as other sludges or leaves a friable scale easy to remove. Compounds of this type, if of honest composition, are effective with many waters and little risk accompanies their use. One great objection to their general use is the opportunity for loading with absolutely worthless materials, such as sawdust and spent tan. The last material named may have a slight value in lessening the density of the scale and in rendering it less difficult to remove, but these features bring spent tan under the second class of compounds.

Sodium fluoride is distinctly a boiler compound and is not suitable for use outside the boiler, as the precipitation of lime and magnesia as fluoride is very imperfect in the cold. It is claimed for sodium fluoride that the reactions with the lime and magnesia compound are almost instantaneous in the boiler, and that the sludge of fluorides produced has no tendency whatever to adhere to the boiler tubes or sheets. It is further asserted that one-fourth of the theoretical amount of sodium fluoride will produce a sludge that will not adhere. This last statement would seem to be question-

able, but from the nature of the sludge produced it may be accepted that less than the theoretical amount will prevent scale formation to any great extent. In cases where the steam must not be alkaline, as in certain manufacturing processes in which steam is used for other than power purposes, sodium fluoride has the advantage of producing steam without any alkaline reaction.

41. The boiler compounds of the second group are of a gelatinous, starchy, or oily nature, and coat or become attached to the surface of the newly formed crystals. Later, when this precipitate is deposited, it has largely lost its cohesive power to cement together and form a hard scale. This scale can be removed as easily as any ordinary sludge by blowing off, or it can be readily removed by means of cleaning tools.

Compounds of this kind are very little used at present, but they are still not infrequently met with among smaller steam plants. They are objectionable, because they thicken and foul the water and coat the boiler with a non-conducting substance that is liable to produce local overheating, causing bagging and burning of tubes or sheets. They are composed of various starchy or gelatinous materials, such as slippery elm, potatoes, starch, fats, or oils, ground bones, hoofs, horns, etc. Their action is due to entrapping mechanically the scale-forming material as it is thrown out of solution with the concentration of the water in the boiler. This gives a sludge to be blown out or a scale, from many waters at least, that is not difficult to remove.

42. *Petroleum* and *kerosene* form the third group of compounds. They assist to some extent in coating the sheets or tubes so that scale will not adhere readily. Crude oil containing large amounts of either readily volatile oils or waxes should not be used. These materials are fed in with the boiler supply, and need to be used with great caution. They, too, are liable to cause local overheating, resulting in bagging and burning. It is claimed by some authorities their use is always dangerous, as they may cause explosions.

While the intention here is simply to refer to this phase of the matter, yet it may be said that both the second and third groups of compounds are held by some boiler authorities to possess a very considerable element of danger. This is largely due to local overheating caused by a deposit of non-conducting materials. Thus, the deposit may prevent the heat of the fire from being carried away by the circulating water and thereby raise portions of the shell or tubes to such an excessive temperature as to cause them to burn or bag; or, on the sudden removal of the non-conducting deposit, the water may be brought in contact with a red-hot section of the metal, which is very severe on the boiler and may even be very dangerous. For these and other reasons, the first group comprises not only the more important compounds, but the ones that have nearly pushed the others out of use in the field where boiler compounds still flourish.

43. Another class of compounds used to some extent consists of *sucrates* or *sugar* in some form. The efficiency of compounds of this kind is due to the increased solubility of the lime compounds.

Molasses, owing to its low cost, is the form in which sugar is usually applied for action on the sulphates or for the neutralization of acids. It is applied either alone or in combination with other softeners, generally sodium carbonate. The molasses dissolves large quantities of lime salts and retards their deposition; and when these salts are deposited, they have little power to adhere to the boiler tubes and shell and can be blown out as sludge.

The chemical action of this kind of compound consists in the formation of a sucrate of lime, which is highly soluble in water. Under the boiler temperature and pressure, the salt first formed, namely, monocalcic sucrate, breaks up into other sucrates that are less soluble in hot than in cold water. Thus, when the boiler is left to cool, the cold water dissolves lime compounds that may have been deposited while the boiler was under steam. The lime salts are dissolved to such an extent that several times the original volume of

water contained in the boiler may be evaporated before it is necessary to blow out the sludge. As in using any other compounds, scale will form if an insufficient quantity is used; also, the boilers will have to be cleaned thoroughly at intervals, depending on the quantity and the quality of water evaporated.

Glycerine has been recommended as a boiler compound. Its effectiveness is due to the solubility of the lime salts in glycerine, which prevents them from forming a hard scale when precipitated. While there is little question as to the efficiency of glycerine, its cost and other considerations render it more a matter of theoretical interest than practical importance.

INTRODUCTION OF COMPOUNDS INTO BOILERS

44. The introduction of compounds into the boiler or the feedwater has frequently resulted in failures or partial failures in the effectiveness of compounds. While the matter is not complicated, it is of the utmost importance that regularity and constancy of feed be under accurate control to secure the benefits possible from the additions.

45. Simple Compound-Introducing Apparatus. In Fig. 13 is shown a simple apparatus for introducing a supply of boiler-compound material in solution into the boiler or the feedwater. The apparatus consists of a reservoir *A* of convenient size attached to a suction pipe *c*. Attached to the reservoir is an upper valve *a* and a bottom valve *b*, to control the admission of the compound. In use, the reservoir is filled with the solution by opening the upper cock, while the lower cock is closed. Cock *a* is then closed and *b* opened, when the suction pulls the compound in with the feedwater. Some degree of regulation of the amount of solution introduced can be secured by adjusting the lower cock.

A still simpler arrangement is to attach by means of a valve on the suction, a pipe that dips into an open vessel containing the compound, the valve serving to control the admission.

The arrangements just outlined serve more or less satisfactorily in plants of small and medium size, but in large plants or those in which the water supply varies greatly at short intervals, such schemes do not satisfactorily perform this work, as the amount of solution fed is not subject to ready regulation; either an excess or a deficiency of reagents is liable to be fed. Furthermore, inattention may allow air to be pulled into the feed-pumps.

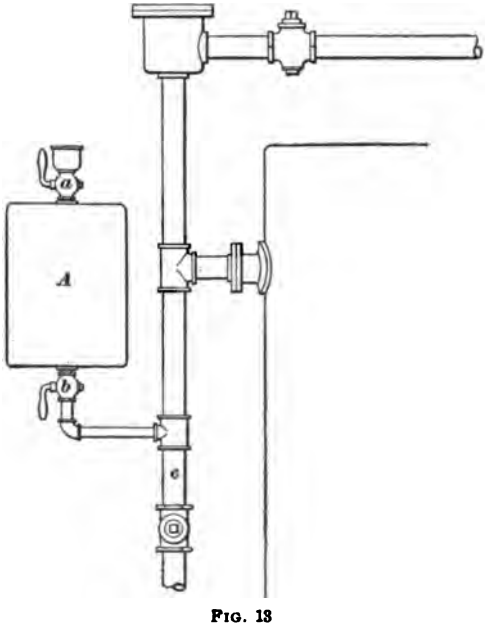


FIG. 13

46. Noey's Apparatus.—The **Noey patented apparatus,** shown in plan and vertical section in Fig. 14, is free from the preceding objections, and is believed to be the best apparatus developed for the introduction of reagents direct to the boiler. It consists of an open tank *a*, preferably of steel, of a size corresponding to the quantity of solution to be introduced into the feedwater. At the bottom, connection is made to the suction of the boiler feed-pumps. A float *b* controls the valve *c*, which is closed in case the water level

gets too low, thus preventing the pumps from sucking air as the tank becomes empty. A small supply pipe furnishes water for dissolving the reagents, which are added to the

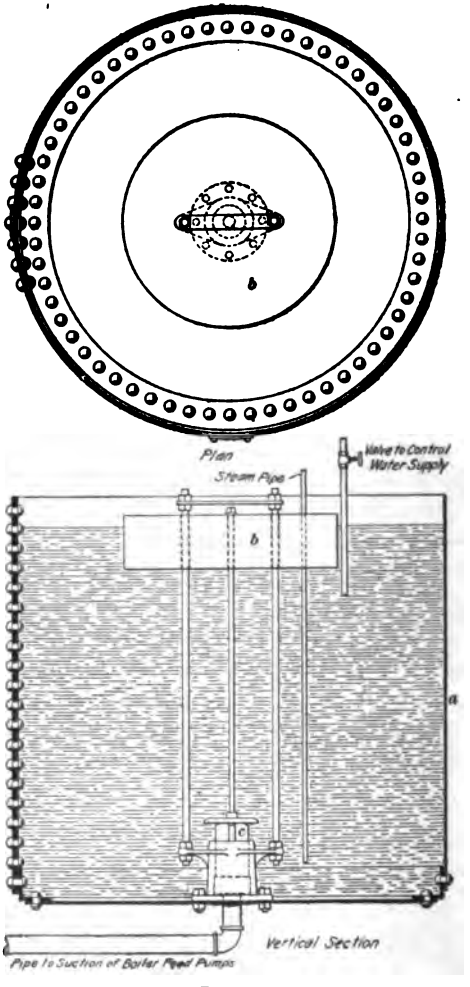


FIG. 14

tank at hourly or other regular intervals, as required. A small steam pipe extends to the bottom of the tank, and the steam serves to heat the water and agitate the solution.

This type of apparatus has the advantage of simplicity and cheapness in construction and operation, gives a positive and regular feed of reagent, and may be readily adjusted to increase or decrease the rate of feed by adjusting the water-supply valve and also the amount of reagent to be added to the tank.

FILTERS AND SOFTENED WATER

CLASSIFICATION AND DESCRIPTION OF FILTERS

47. Filters are frequently used with water-softening apparatus, especially apparatus of the intermittent type. They are also extensively used for the purification of municipal water supplies, and for this purpose are almost univerally of the *open-sand type*. Here, however, only the application of filters in connection with water-softening plants is to be considered. In all cases, filters serve to remove suspended matter and a large percentage of bacteria, the removal of the latter being their most important function in municipal water supply.

48. Filter Medium.—The **filter medium** may be composed of various substances, such as gravel, crushed quartz, sand, coke, charcoal, linen, felt, excelsior, sponge, etc. The standard filter is, however, almost universally composed of silica, as sand, gravel, or quartz, and frequently the three in combination. The material in a filter is arranged like that in sand or gravel banks and the bottoms of streams; that is, coarse material on the bottom, then successive layers of less coarse material, with fine sand on the top.

49. Classification of Filters.—Two general classes of filters are in common use, namely, the *pressure* and the *gravity*. As the names imply, the former is operated under pressure, while the latter is an open-top filter with only the head of water on the filter to force the water through.

50. Gravity Filter.—In Fig. 15 is shown a **gravity filter** of the Continental-Jewell type, with a mechanical

agitation device. This filter consists of a wooden tank *a* of sufficient capacity, the bottom of which is covered with Portland cement. Resting on the bottom of the tank is a system of underdrains *b* made up of pipes with perforated nozzles. These pipes are connected to a single manifold *c* extending

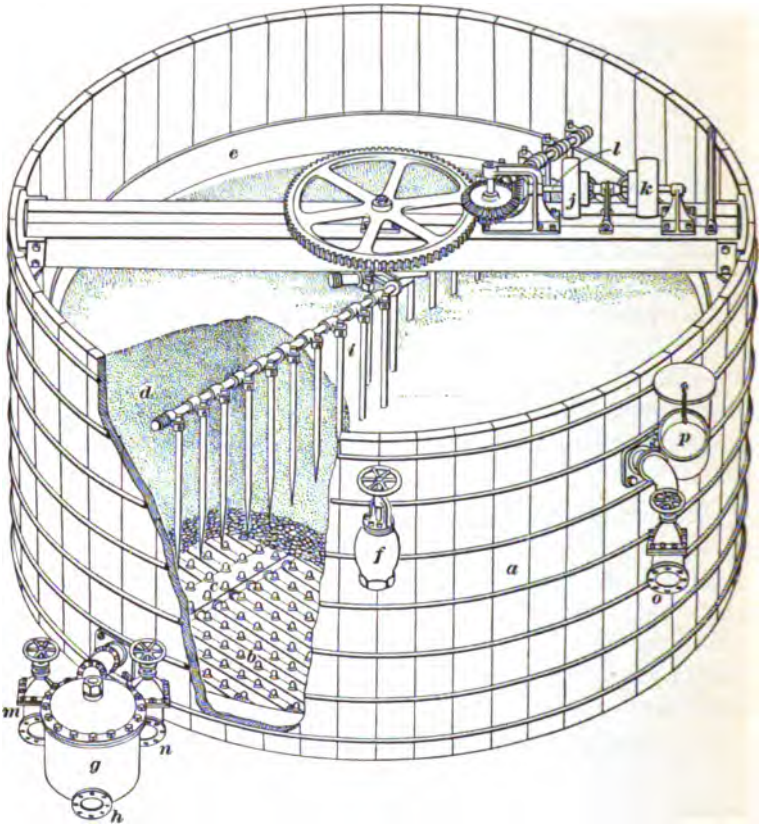


FIG. 15

from one side of the tank to the other. Directly over the underdrains is a layer of gravel, and on top of this layer is a thick bed *d* of clean sand. A trough *e* extending entirely around the inside of the tank serves as an overflow to carry off the water used in cleaning the filter.

The operation of the filter is as follows: Raw water enters on top of the filtering bed through valve *f*, by which the rate of entry is controlled, and percolates through the filtering material, passing into the underdrain and to the manifold *c*. It then passes to a regulator *g* that contains a float valve designed to insure a uniform rate of filtration. The filtered water then passes to its destination through a pipe bolted to flange *h*.

To clean the filter bed thoroughly, the sand of which it is composed is stirred up by means of a rake *i*. This rake is driven through gearing and suitable shafting, and by either a steam engine or an electric motor. To permit a ready reversal of the direction in which the rake rotates, driving is accomplished from an open and a crossed belt placed on the pulleys *j* and *k*, respectively. Reversal is accomplished by unclutching the one pulley from the driving shaft *l* and clutching the other pulley to it. Friction clutches are used for this purpose. The valves *m* and *n* both connect with the manifold *c*. The valve *m* has a pipe connection to an air compressor, and the valve *n* has a connection to the raw-water supply. The valve *o* connects on one side with the trough *e*, and on the other, with the sewer. The condition of the water can be ascertained through a handhole *p*, which has a removable cover. Cleaning of the filter bed is accomplished by alternately admitting raw water and compressed air through the valves *n* and *m*, respectively, the valve *f* being closed and *o* opened; at the same time, the sand bed is agitated by rotating the rake *i* first in one direction and then in the reverse direction.

51. The Reisert Filter.—The **Reisert filter**, shown in Fig. 16, has had extensive use in Europe, but so far it has not been adopted in the United States. It has been used in connection with both the intermittent and the continuous apparatus, as well as alone to filter water that does not need to be softened. The filter is automatic and continuous in operation and is constructed essentially as shown.

The water enters the space *b* through the pipe *a*, and flowing down and through the annular pipe, it overflows into *c*.

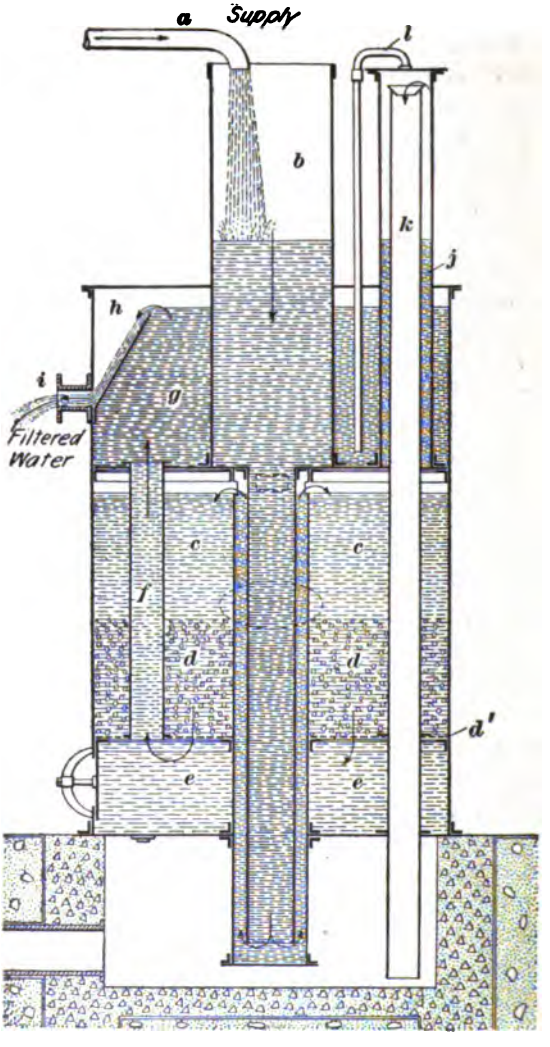


FIG. 16

It then passes through the sand-and-gravel filter *d* supported on the perforated plate *d'*. From the filter, the water enters

the reservoir, or chamber *e*, then passes through *f* to *g*, and flowing over the division plate *h*, finally passes through *i* to storage tanks or reservoirs. The head of water in the space *b* gives the pressure to force the water through the filter and out through *i*. As the filter *d* becomes clogged, the water gradually rises in *b*, maintaining the same level in the pipe *j*, which is connected with *c*, until it finally overflows into the annular pipe *k*, thus creating a siphon that serves to reverse the direction of flow. The pipe *k* extends down through the entire apparatus and discharges into the sewer below *e*. It thus forms the long leg of a siphon, and will pull water from the space *e*. This water passes up through *d* and cleanses the filter. The flow continues in this reverse direction until the water in *b* falls below the open end of the small pipe *l*, when air is sucked into *k*, thus stopping the flow of water in the siphon. The filter then resumes its regular working until the water rises to the top of *k*, when the cleansing is automatically repeated.

52. Pressure Filters.—The pressure from mains or pumps generally operates **pressure filters**, which are closed, steel tanks. However, in the case of softening plants, they are operated as a rule by the pressure from reaction tanks placed at a higher level than are the filters. Pressure filters are made in almost any size units, but as a rule not above a daily capacity of 500,000 gallons. Where a larger volume of water has to be filtered, rather than greatly increase the size of individual filters, a sufficient number of filters are installed to handle it. In all cases, provision is made for cleaning filters as required, the interval for this depending on the amount and character of the precipitate or other matter in suspension. Cleaning is generally accomplished by a reversal of the flow of water. The water, under sufficient pressure, enters below the filtering medium and is distributed over the entire area of the filter, to insure thorough cleansing. This distribution is accomplished by various devices in different types of filters. Air under pressure is sometimes used in connection with the water.

Details differ as to filling, methods of cleaning, etc., and the filters may be either horizontal or vertical. The pressure filter is in all essential principles the same as the open gravity filter, but is contained in a steel cylinder to withstand whatever pressure is required, suitable provision being made for

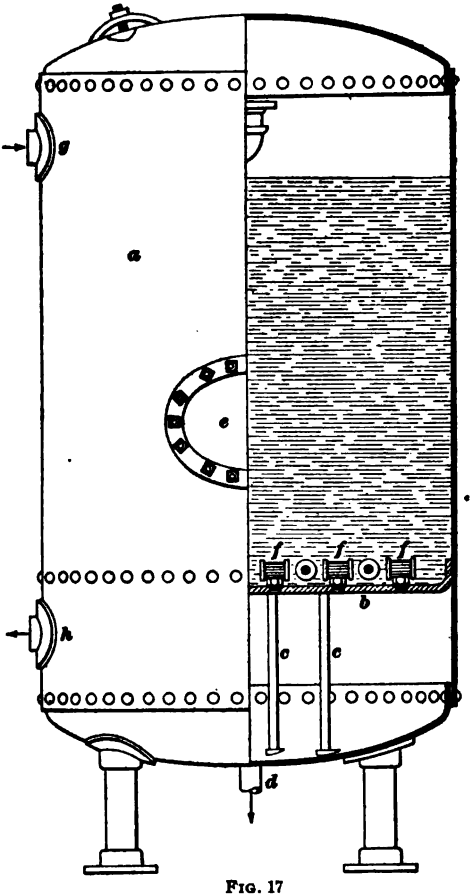


FIG. 17

reversing the flow of water for cleaning. For water-softening plants of large size, in which either gravity or pressure filters are employed, sufficient filters are provided so that one or more may be cleaned without interrupting the supply.

53. In Fig. 17 is shown a pressure filter and the principle on which practically all filters of this type are constructed. This pressure filter is of the vertical type and consists of a steel shell *a* that is closed by dished heads and is fitted with a false bottom *b* braced by uprights *c* to support the filter bed. The open space below the false bottom serves as a settling chamber and has a pipe *d* leading to the sewer and fitted with a valve. Access to the interior is provided by a removable cover *e*. Fitted to the upper side of the bottom *b* are a large number of T-shaped, hollow underdrains *f*. These underdrains have five slots cut into them and open into the settling chamber. The filtering bed, not shown in the figure, consists of a layer of gravel placed over the underdrains, on top of which is a thick bed of clean sand.

In operation, the water enters the top of the filter at *g*, passes through the filter bed into the settling chamber, and then passes out to its destination through *h*. The inlet and outlet pipes are so connected by pipes and valves that, for cleaning the filter bed, the water under pressure can enter at *h* and leave through *g*, passing through a by-pass pipe to the sewer.

54. Filter Presses.—Another form of filter that has been used extensively, especially in Europe, is the **filter press.** It is not believed that this type of filter is used at present in the United States for filtering softened water. Its use is confined principally to certain chemical and allied industries, but it is thought to be of sufficient general interest to warrant illustration. Fig. 18 shows the ordinary type of filter press. It consists of a series of chambers *a* formed of removable screens *b*, which are covered by either cloths or paper, as the one shown at *c*, and made tight by a screw.

A circular press with a hollow shaft for introducing the unfiltered water has also been used.

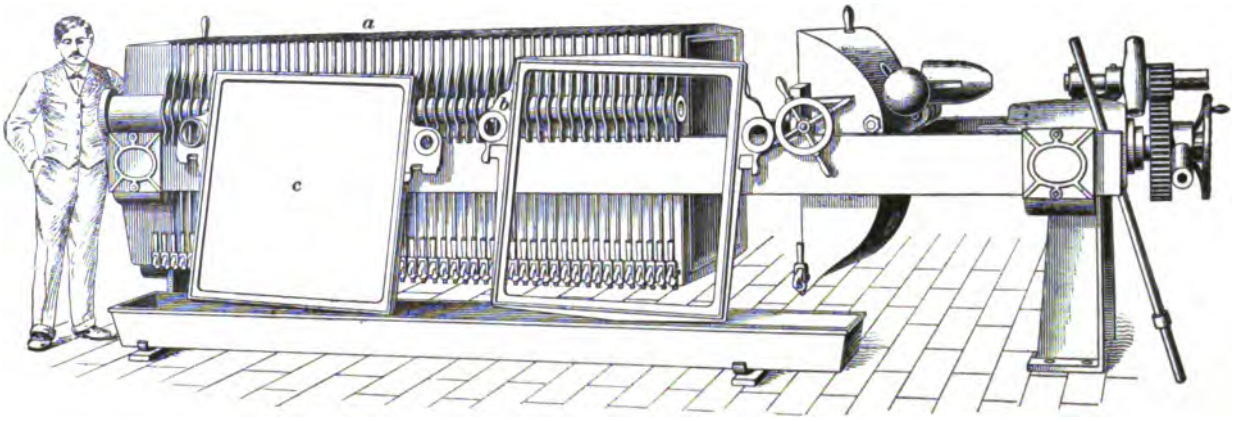


FIG. 18

USES OF SOFTENED WATER

55. Without doubt the greatest use of treated water is to produce steam for power purposes. Many other uses, however, on a large scale are made of treated water, principally in the chemical and allied industries. One of the most important uses is in dyeing and bleaching in the various textile industries. Waters containing high percentages of salts injuriously affect colors by causing reactions with the coloring matter, thus altering them greatly or producing compounds that precipitate on the goods. Iron compounds, as well as lime salts and organic matter, are especially objectionable.

Another important use of softened water is in soap works and laundries. In the former, the alkalies used are wasted by reacting with the salts in the water; thus, the quality of the soap is injuriously affected and the yield reduced. For laundry and household purposes, it is conservatively estimated that about 1 pound of ordinary soap is consumed for each degree of hardness per 1,000 gallons, due to the fact that lime and magnesia salts form insoluble soaps. This not only wastes the soap, but the resulting curd adheres to the fabrics, making a harsh product that is frequently of a yellowish tint and from which it is difficult to remove dirt thoroughly.

56. Taking the preceding estimate of soap wasted, it will be readily seen that economy would result from softened water when using even a moderately hard supply. In order to illustrate, a municipal supply having a hardness of 10°, which is quite common, will be considered. An approximate average consumption per capita is 80 gallons per day, or in a city of 100,000 inhabitants, 8,000,000 gallons would be needed. This quantity of water could be softened from 10° to 6° at a cost of about 1 cent per thousand gallons, and less in most cases, or at a total cost of $80 per day for chemicals. Assume that one-fourth of an average city supply, which is probably below the average, is used with soap or for purposes for

which a hard water is equally detrimental. This, then would mean 2,000,000 gallons. Allowing the preceding estimate of 1 pound per thousand gallons for each degree of hardness, it will be seen that 8,000 pounds of soap is wasted per day. At 5 cents per pound, it would mean that $400 worth of soap is used. Of course, much of the domestic supply is softened with soda, borax, etc., but at a much greater cost per unit than all the water could be treated for in a softening plant. For a population of 100,000, a safe saving would be at least half the foregoing, or $200 per day. The difference between this and the cost of chemicals, or $80, would represent nearly $45,000 per year, which would go a very long way toward capitalizing and operating a softening plant.

While it would probably not pay to soften a water having a hardness of 10°, there is no doubt that many municipal supplies would show a handsome profit on the cost of softening, to say nothing of the greater healthfulness and cleanliness that would result from using such a cleansed water. Several towns of considerable size soften their water, and there would seem to be no doubt that the next few years will see many city supplies having a hardness of 10° to 15° or more softened, with excellent rewards for the outlay.

57. A softened water is thoroughly wholesome for drinking and culinary purposes, and, aside from economy, offers many advantages for domestic use. It is not necessarily free from bacteria, but in every case where present, especially pathogenic organisms, the number is greatly reduced, and with proper care, all ordinary bacteria may be almost completely eliminated. Soft water is much to be preferred for all other domestic purposes. In cooking, a hard water deposits salts on the surface of meats, vegetables, etc., making the tissues harder and less nutritive. The curd, or scum, produces a greasy deposit in utensils, sinks, and pipes, and in some cases adds to the difficulty of the sewerage problems.

58. In tanning, after the unhairing process by the use of lime, waters carrying bicarbonates cause a precipitation

of the carbonate in the tissues of the hides. This precipitation prevents the ready absorption of tannin in the cells, and thus delays and impairs the tanning process.

In paper making, a soft or at least clear supply of water is of great advantage in all cases and an absolute essential in making many of the finer grades. In sizing, hard waters interfere with the precipitation of the resin by the alum, some of the resin combining with the lime to form a curdy soap that deteriorates the sizing process.

In many other industries, soft water is either essential or adds greatly to the quality of the product.

For photographic purposes, many waters have to be purified. This is done on a small scale by adding soda ash, sodium bicarbonate, alum, or caustic lime to a barrel or tank of the water and allowing it to settle. Matter in suspension or solution is inimical to proper developing or printing. In large photographic manufacturing plants, if a naturally pure and clear water is not available, the water is softened on a commercial scale for the preparation of the various developing solutions, plates, films, etc.

59. The brewing industry is about the only one for which a softened water is not suitable. Here, a water containing calcium sulphate (gypsum) is essential, or, rather, gives results that are much superior. This fact is so well recognized that if the water does not naturally contain the salt, it is added. The celebrated English, Burton-on-Trent ales and beers owe their reputation to the large percentage of gypsum contained in the water used. It is claimed that this water has more than 60 grains of gypsum per gallon. Gypsum, however, is the only salt that is of advantage, organic matters and the usual hardening salts being objectionable. If the latter are present in more than small amounts, the water is softened to remove the bicarbonate and other salts, leaving the sulphates in solution, and the gypsum required is then added. In this way, exact control of the amount may be had, the supply is not subject to variations, and all injurious matters may be removed.

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